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SLUDGE WASHING AND DEMONSTRATION OF THE DWPF FLOWSHEET IN THE SRNL SHIELDED CELLS FOR SLUDGE BATCH 6 QUALIFICATION

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EXECUTIVE SUMMARY

Prior to initiating a new sludge batch in the Defense Waste Processing Facility (DWPF), Savannah River National Laboratory (SRNL) is required to simulate this processing, including Chemical Process Cell (CPC) simulation, waste glass fabrication, and chemical durability testing. This report documents this simulation for the next sludge batch, Sludge Batch 6 (SB6). SB6 consists of Tank 12 material that has been transferred to Tank 51 and subjected to Low Temperature Aluminum Dissolution (LTAD), Tank 4 sludge, and H-Canyon Pu solutions. Following LTAD and the Tank 4 addition, Liquid Waste Operations (LWO) provided SRNL a 3 L sample of Tank 51 sludge for SB6 qualification. Pu solution from H Canyon was also received. SB6 qualification included washing the sample per LWO plans/projections (including the addition of Pu from H Canyon), DWPF CPC simulations, waste glass fabrication (vitrification), and waste glass characterization and chemical durability evaluation.

The following are significant observations from this demonstration.

- Sludge settling improved slightly as the sludge was washed.

- SRNL recommended (and the Tank Farm implemented) one less wash based on evaluations of Tank 40 heel projections and projections of the glass composition following transfer of Tank 51 to Tank 40.

- Thorium was detected in significant quantities (>0.1 wt % of total solids) in the sludge. In past sludge batches, thorium has been determined by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS), seen in small quantities, and reported with the radionuclides. As a result of the high thorium, SRNL-AD has added thorium to their suite of Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) elements.

- The acid stoichiometry for the DWPF Sludge Receipt and Adjustment Tank (SRAT) processing of 115%, or 1.3 mol acid per liter of SRAT receipt slurry, was adequate to accomplish some of the goals of SRAT processing: nitrite was destroyed to below 1,000 mg/kg and mercury was removed to below the DWPF target with 750 g of steam per g of mercury. However, rheological properties did not improve and were above the design basis.

- Hydrogen generation rates did not exceed DWPF limits during the SRAT and Slurry Mix Evaporator (SME) cycles. However, hydrogen generation during the SRAT cycle approached the DWPF limit.

- The glass fabricated with the Tank 51 SB6 SME product and Frit 418 was acceptable with respect to chemical durability as measured by the Product Consistency Test (PCT). The PCT response was also predictable by the current durability models of the DWPF Product Composition Control System (PCCS). It should be noted, however, that in the first attempt to make glass from the SME product, the contents of the fabrication crucible foamed over. This may be a result of the SME product's

REDOX (Reduction/Oxidation – $Fe^{2+}/\Sigma Fe$) of 0.08 (calculated from SME product analytical results).

The following are recommendations drawn from this demonstration.

- In this demonstration, at the request of DWPF, SRNL caustic boiled the SRAT contents prior to acid addition to remove water (to increase solids concentration). During the nearly five hours of caustic boiling, 700 ppm of antifoam was required to control foaming. SRNL recommends that DWPF not caustic boil/concentrate SRAT receipt prior to acid addition until further studies can be performed to provide a better foaming control strategy or a new antifoam is developed for caustic boiling.

- Based on this set of runs and a recently completed demonstration with the SB6 Waste Acceptance Product Specifications (WAPS) sample, it is recommended that DWPF not add formic acid at the design addition rate of two gallons per minute for this sludge batch. A longer acid addition time appears to be helpful in allowing slower reaction of formic acid with the sludge and possibly decreases the chance of a foam over during acid addition.

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LIST OF ABBREVIATIONS

AD	Analytical Development
AR	Aqua Regia digestion
ARG-1	Analytical Reference Glass – 1
ARM	Approved Reference Material
CPC	Chemical Process Cell
CVAA	Cold Vapor Atomic Absorption
C-XRD	Contained X-Ray Diffraction
DWPF	Defense Waste Processing Facility
EA	Environmental Assessment
FAVC	Formic Acid Vent Condenser
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma-Mass Spectroscopy
LRM	Low Activity Test Reference Material
LTAD	Low Temperature Aluminum Dissolution
LWO	Liquid Waste Operations
MA	Mixed Acid
MAR	Measurement Acceptability Region
MWWT	Mercury Water Wash Tank
NIST	National Institute of Standards and Testing
PCCS	Product Composition Control System
PCT	Product Consistency Test
PF	Peroxide Fusion digestion
R&D	Research and Development
REDOX	Reduction/Oxidation ($Fe^{2+}/\Sigma Fe$)
SB6	Sludge Batch 6
SME	Slurry Mix Evaporator
SMECT	Slurry Mix Evaporator Condensate Tank
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TIC	Total Inorganic Carbon
TTQAP	Task Technical and Quality Assurance Plan
WAPS	Waste Acceptance Product Specification

1.0 Introduction

Sludge Batch 6 (SB6) consists of Tank 12 material that has been transferred to Tank 51 and subjected to Low Temperature Aluminum Dissolution (LTAD), Tank 4 sludge, and H-Canyon Pu solutions. Following LTAD and the Tank 4 addition, Liquid Waste Operations (LWO) provided the Savannah River National Laboratory (SRNL) a 3 L sample of Tank 51 sludge for SB6 qualification.[1] Pu solution from H Canyon was also received. SB6 qualification included washing the sample per LWO plans/projections (including the addition of Pu from H Canyon), DWPF Chemical Process Cell (CPC) simulations, waste glass fabrication (vitrification), and waste glass characterization and chemical durability evaluation.

This report documents:

- The washing (addition of water to dilute the sludge supernate) and concentration (decanting of supernate) of the SB6 - Tank 51 qualification sample to adjust sodium content and weight percent insoluble solids to Tank Farm projections.
- The performance of a DWPF CPC simulation using the washed Tank 51 sample. The simulations included a Sludge Receipt and Adjustment Tank (SRAT) cycle, where acid was added to the sludge to destroy nitrite and reduce mercury, and a Slurry Mix Evaporator (SME) cycle, where glass frit was added to the sludge in preparation for vitrification. The SME cycle also included replication of five canister decontamination additions and concentrations. Processing parameters were based on work with a non radioactive simulant.[2]
- Vitrification of a portion of the SME product and characterization and durability testing (as measured by the Product Consistency Test (PCT)) of the resulting glass.
- Rheology measurements of the initial slurry samples and samples after each phase of CPC processing.

This work was controlled by a Task Technical and Quality Assurance Plan (TTQAP)[3], and analyses were guided by an Analytical Study Plan[4]. This work is Technical Baseline Research and Development (R&D) for the DWPF.

2.0 Experimental Procedure

The Experimental Procedure section is divided into several subsections that reflect the major parts of this task: sludge washing, CPC simulations, and glass fabrication and durability testing. An additional subsection, presented first, describes the analytical methods utilized for each of these tasks.

2.1 Analytical Methods

Described below are the methods and techniques used to generate the analytical data presented in this report.

2.1.1 *As-Received, SRAT Receipt and SRAT Product Characterization*

Eight separate aliquots of the slurry for each type of sample were digested, four with HNO_3/HCl (aqua regia[5]) in sealed Teflon® vessels and four in Na_2O_2 (alkali or peroxide fusion[6]) using Zr crucibles. Due to the use of Zr crucibles and Na in the peroxide fusions, Na and Zr cannot be determined from this preparation. Additionally, other alkali metals, such as Li and K, and alkaline earth metals, such as Ca, that may be contaminants in the Na_2O_2 are not determined from this preparation. Three Analytical Reference Glass – 1[7] (ARG-1) standards were digested along with a blank for each preparation. The ARG-1 glass allows for an assessment of the completeness of each digestion. Each aqua regia digestion and blank was diluted to100 mL or 250 mL with de-ionized water and submitted to Analytical Development (AD) for inductively coupled plasma – atomic emission spectroscopy (ICP-AES) analysis, inductively coupled plasma – mass spectrometry (ICP-MS) analysis of masses 81-209 and 230-252, and cold vapor atomic absorption (CVAA) analysis for Hg. Equivalent dilutions of the peroxide fusion digestions and blank were submitted to AD for ICP-AES analysis.

The aqua regia SRAT Receipt and SRAT Product solutions from digestion contained undissolved solids. A portion of the solids were recovered by filtration from one of the SRAT Receipt samples, submitted to Contained X-Ray Diffraction (C-XRD) for analysis and identified as boehmite. The undissolved solids in the SRAT Product digestion solutions were not analyzed but assumed to be boehmite as well.

The elemental concentrations reported are either a combination of both digestion methods and an average of eight data points or an average of four data points from one digestion method. The aqua regia and peroxide fusion methods agreed well for most major elements (greater than 0.5 wt% of total solids), except Al. See Click et al., SRNL-STI-2010-00259, Revision 0, for a statistical comparison of digestion data.

2.1.2 *Glass Dissolution Methods and Analyses*

To support compositional analysis, a portion of the SB6 Qualification Glass had to be dissolved. In order to enhance dissolution, approximately 4 g of the glass was crushed and ground using agate cups, balls and caps in a mechanical pulverizing mixer mill. The glass was sieved and only the portion that passed through a 200 mesh (<75 μm) brass sieve was used for the dissolutions. Weighed amounts (nominally 0.25 g) of the crushed glass were then dissolved remotely by two different methods to ensure that all the elements of interest were dissolved and could be analyzed. The two methods were a sodium peroxide fusion at 675 °C followed by a HNO_3 uptake, and an acid dissolution in sealed vessels at 115 °C using a combination of HF, HCl, and HNO_3 acids. Boric acid was added to this latter dissolution method to complex excess fluoride. The solutions of the dissolved glass were diluted to known volumes so that approximately 15 mL aliquots could be safely removed from the Shielded Cells without exposing personnel to excess radiation. Four aliquots of the crushed SB6 Qualification Glass were dissolved by each technique. The aliquots were then submitted to AD, where they were analyzed by ICP-AES, radioactive counting techniques, and by Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS). Concurrent with each set of dissolutions in the Shielded Cells, three samples of ARG-1 were also dissolved to determine if the dissolutions were complete and the resulting analyses accurate. With each set of samples sent to AD, two samples of a multi-element standard containing known concentrations of Al, B, Fe, Li, Na, and Si were also submitted.

2.1.3 *Supernate Sample Preparation For Analysis*

A portion of the well-mixed sludge slurry was filtered through a 0.45 µm porosity filter. Portions of the filtered supernate were diluted with de-ionized distilled water or nitric acid to reduce the sample activity and allow removal from the Shielded Cells for chemical analysis. All sample preparations of the filtered supernate samples were conducted in quadruplicate. A blank was prepared along with the sample dilutions.

2.1.4 *Weight Percent Solids and Density Measurements*

The densities of the filtered supernate and the well-mixed slurry sample were measured in the Shielded Cells using calibrated plastic tubes with a nominal volume of ~8.25 mL. The density measurements were conducted in quadruplicate on each phase of the sample. The weight percent total solids in the slurry sample were measured in the Shielded Cells using a conventional drying oven at 110 °C. The sample was dried until repeated weights indicated no further loss of water. The weight percent dissolved solids in a sample of the filtered supernate were measured in the same manner. All weight percent solids measurements were made in quadruplicate. The weight percent insoluble solids and weight percent soluble solids in the slurry sample were calculated using the equations shown below.

Equation 1

$$W_{is} \frac{W_{ts} - W_{ds}}{1 - W_{ds}}$$

Equation 2

$$W_{ss} = W_{ts} - W_{is}$$

where:

W_{is} = weight fraction of insoluble solids in the slurry
W_{ss} = weight fraction of soluble solids in the slurry
W_{ts} = weight fraction of total solids in the slurry
W_{ds} = weight fraction of dissolved solids in the filtered supernate

Thus:

Wt% dissolved solids = (wt dissolved solids/wt of supernate) x 100
Wt% total solids = (wt total solids/wt of total slurry) x 100
Wt% insoluble solids = (wt insoluble solids/wt of total slurry) x 100
Wt% soluble solids = (wt of dissolved solids/wt of total slurry) x 100

2.1.5 *Rheology*

Rheological properties of radioactive samples are determined using a Haake M5/RV30 rotoviscometer. The M5/RV30 is a Searle sensor system, where the bob rotates and the cup is fixed. The torque and rotational speed of the bob are measured. Heating/cooling of the cup/sample/bob is through the holder that holds the cup. The shear stress is determined from the torque measurement and is independent of the rheological properties. Conditions that impact the measured torque are; slip (material does not properly adhere to the rotor or cup), phase separation (buildup of liquid layer on rotor), sedimentation (particles settling out of the shearing zone), homogeneous sample (void of air), lack of sample (gap not filled), excess sample (primarily impacts rheologically thin fluids), completely filling up the void below the bob (air buffer that is now filled with fluid) and Taylor vortices. The first five items yield lower stresses and the last three add additional stresses. The shear rate is geometrically determined using the equations of change (continuity and motion) and is that for a Newtonian fluid. This assumption also assumes that the flow field is fully developed and the flow is laminar. The shear rate can be calculated for non-Newtonian fluid using the measured data and fitting this data to the rheological model or corrected as recommended by Darby[8]. In either case, for shear thinning non-Newtonian fluids typical of Savannah River Site (SRS) sludge wastes, the corrected shear rates are greater than their corresponding Newtonian shear rates, resulting in a thinner fluid. Correcting the flow curves will not be performed in this task, resulting in a slightly more viscous fluid.

The bob typically used for measuring tank sludge or SRAT product is the MV I rotor. For SME product, the MV II rotor is used to perform the measurements, due to the larger frit particles that are present in the SME product. The MV II has a larger gap to accommodate the larger frit particles. The shape, dimensions, and geometric constants for the MV I and MV II rotors are provided in Table 2-1.

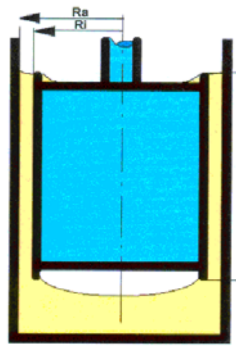
Prior to performing the measurements, the rotors and cups are inspected for physical damage. The torque/speed sensors and temperature bath are verified for functional operability using a bob/cup combination with a National Institute of Standards and Technology (NIST) traceable Newtonian oil standard, using the MV I rotor. The resulting flow curves are then fitted as a Newtonian fluid and this calculated viscosity must be within ± 10% of the reported NIST viscosity at a given temperature for the system to be considered functionally operable. A N10 oil standard was used to verify system operability prior to the sludge measurements.

The flow curves for the sludge are fitted to the down curves using the Bingham Plastic rheological model, Equation 3, where τ is the measured stress (Pa), τ_o is the Bingham Plastic yield stress (Pa), μ_∞ is the plastic viscosity (Pa·sec), and $\dot{\gamma}$ is the measured shear rate (sec^{-1}). During all these measurements, the sample remained in the cup for the 2nd measurement, due to the sample availability.

Equation 3 $$\tau = \tau_o + \mu_\infty \dot{\gamma}$$

Table 2-1. MV I and MV II Rotor Specifications and Flow Curve Program

Rotor Design



Dimensions and Flow Curve Program

Rotor Type	MV I	MV II
Rotor radius - R_i (mm)	20.04	18.40
Cup Radius - R_a (mm)	21.0	21.0
Height of rotor -L (mm)	60	60
Sample Volume (cm^3) minimum	40	55
A factor (Pa/%torque)	3.22	3.76
M factor (s^{-1}/%RPM)	11.7	4.51
Shear rate range (s^{-1})	0 – 600	0 – 300
Ramp up time (min)	5	5
Hold time (min)	1	1
Ramp down time (min)	5	5

2.2 <u>Sludge Washing</u>

The as-received sludge was placed into a 4-L glass vessel. The vessel was fitted with an agitator shaft to facilitate mixing. The vessel had volume graduations to aid in settling observations and decant/addition volumes. A photograph of the washing vessel is shown in Figure 2-1. Prior to washing, a subsample was taken for characterization.

Figure 2-1. Photograph of Sludge Batch 6 Washing Vessel

In past sludge batches, a high density polyethylene bottle was used, and the entire vessel would be weighed before and after additions and decants. A glass vessel was utilized in this sludge batch to better evaluate sludge settling. Therefore, because of size and risk of breakage, washing was predominantly done by volume. However, masses of additions and decants were recorded.

Washing (addition and decant amounts) followed Tank Farm plans of November 10, 2009.[9] An excerpt of this spreadsheet is given in Appendix A. SRNL washing and decant amounts were determined by simple ratios:

$$\text{SRNL Addition (decant) Volume} = \text{Tank Farm Addition (Decant) Volume} \times \frac{\text{SRNL Sludge/Slurry Volume}}{\text{Tank Farm Sludge/Slurry Volume}}$$

SRNL washing, decant amounts, and sample amounts are given in Appendix B.

Supernate was characterized (elementals and anions) following each decant. Slurry samples were taken after Decants C and F for a more thorough characterization (aqua regia digestions, weight % total solids). Following the addition of Wash I, a supernate sample was taken. Based on elemental and anion results (primarily sodium), SRNL recommended that washing be stopped, and LWO concurred. SRNL decanted as much supernate as possible (Decant I) to maximize the amount of insoluble solids to be sent to DWPF. Rheological properties of the resultant sludge were measured to ensure the sludge met the design basis limits (supernate would have been added back to lower yield stress and consistency if necessary). The slurry following Decant I became the SB6 Qualification SRAT receipt material.

Sludge level was periodically recorded during settling after wash water additions to provide a semi-qualitative assessment of settling behavior throughout washing.

2.3 Chemical Process Cell Simulation

This section describe the DWPF CPC simulations using the SRNL-washed Tank 51 Sludge Batch 6 sample. The simulations were performed in the SRNL Shielded Cells.

DWPF simulations (SRAT and SME cycles) using the SRNL washed Tank 51 SB6 qualification sample were conducted following procedures in the Environmental and Chemical Process Technology Research Programs Section procedure manual.[10] A summary of each cycle is presented in Table 2-2. At the request of the DWPF, the receipt sample was concentrated from 9.9 wt % insoluble solids to approximately 12 wt % total solids by boiling prior to acid addition.

Table 2-2. Summary of SB6 Qualification CPC Processing

SRAT Cycle	SME Cycle
• Acid Calculation • Concentration (boiling) to raise insoluble solids to 12 wt % • Cooling to approximately 80 °C for sampling • Heating to 93 ºC • Addition of nitric and formic acids per acid calculation • Heat to boiling • Concentration (water removal) to a target wt% total solids • Reflux to obtain a total time at boiling of 35 hours at a DWPF boil-up rate of 5,000 lb steam/h	• Addition and removal of water to simulate addition and removal of water from the decontamination of 5 glass canisters • Addition of frit and dilute formic acid in two batches to target 34% waste loading • Concentration (water removal) to target 45-50 wt% total solids.

The SB6 qualification CPC processing was performed using a vessel designed to process one liter of sludge. The SRAT rig was assembled and tested in the SRNL Shielded Cells Mockup area and placed into the Shielded Cells fully assembled. A detailed description of the SRAT rig and testing of the rigs can be found in reference number 11. Two rigs were ultimately needed after a significant foam-over of sludge in the first rig. The intent of the equipment is to functionally replicate the DWPF processing vessels. The glass kettle is used to replicate both the SRAT and the SME, and it is connected to the SRAT Condenser and the Mercury Water Wash Tank (MWWT). Because the DWPF Formic Acid Vent Condenser (FAVC) does not directly impact SRAT and SME chemistry, it is not included in SRNL Shielded Cells CPC processing. Instead, a simple “cold finger” condenser is used to cool offgas to approximately 20 °C below ambient to remove excess water before the gas reaches the gas chromatograph for characterization. The Slurry Mix Evaporator Condensate Tank (SMECT) is represented by a sampling bottle that is used to remove condensate through the MWWT. For the purposes of this paper, the condensers and wash tank are referred to as the offgas components. A sketch of the experimental setup is given as Figure 2-2.

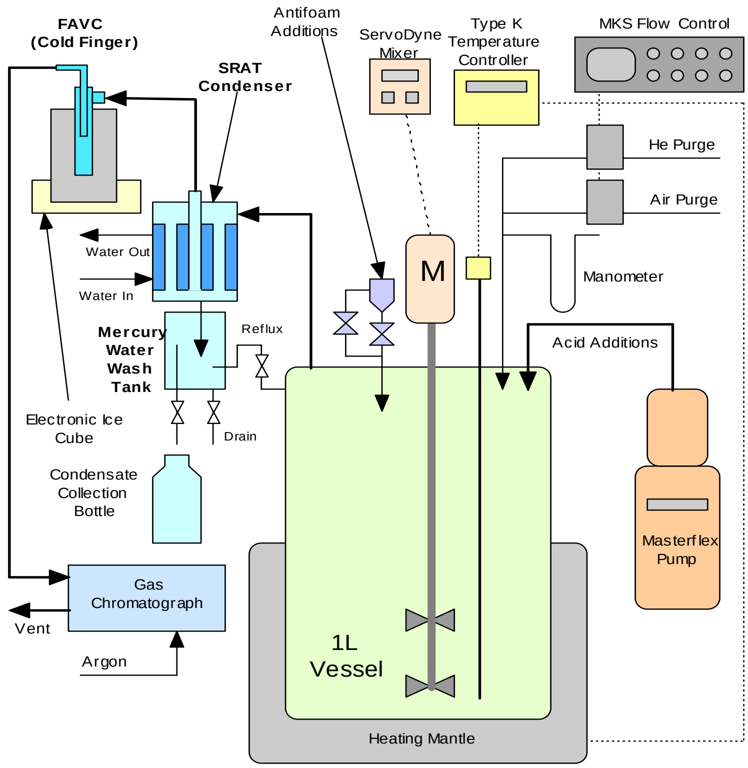


Figure 2-2. Schematic of SRAT Equipment Set-Up

Helium was introduced at a concentration of 0.5% of the total air purge as an inert tracer gas so that total amounts of generated gas and peak generation rates could be calculated. Off-gas concentrations of hydrogen, oxygen, nitrogen, nitrous oxide, and carbon dioxide concentrations were measured during the experiments using in-line instrumentation (an Agilent 3000 series micro GC). Helium was introduced at a concentration of 0.5% of the total air purge as an inert tracer gas so that total amounts of generated gas and peak generation rates could be calculated. During the runs, the kettle was visually monitored to observe reactions that were occurring to include foaming, air entrainment, rheology changes, loss of heat transfer capabilities, and offgas carryover. Observations were recorded in laboratory notebooks[12, 13] and are discussed in Sections 3.2.1 (SRAT cycle) and 3.2.2 (SME cycle).

Concentrated nitric acid (50-wt%) and formic acid (90-wt%) were used to acidify the sludge and perform neutralization and reduction reactions during processing. The amounts of acid to add were determined using the existing DWPF acid addition equation in the 6/1/07 version of the SRNL acid calculation spreadsheet[14]. The split of the acid was determined using the latest Reduction/Oxidation (REDOX) equation.[15] To account for the reactions and anion destructions that occur during processing, assumptions about nitrite destruction, nitrite-to-nitrate conversion, and formate destruction were made based on results from SB6 simulant CPC testing. Acid stoichiometry and reflux time were also based on CPC processing of SB6 simulant sludge slurry.[2]

2.4 Glass Fabrication and PCT

2.4.1 *Glass Fabrication*

There were two attempts at glass fabrication. In the first attempt, approximately 100 g of SME product (SRAT product with Frit 418) was placed in a 95% platinum/5% gold crucible and dried overnight at 110 °C. After drying, the material was then placed in a room temperature furnace which heated at 5 °C per min to a melting temperature of 1150 °C. The sample was held at 1150 °C for four hours. Unfortunately, the contents of the crucible foamed over during this melting process. The REDOX ($Fe^{2+}/\Sigma Fe$) was calculated to be 0.08 based on the current REDOX equation using the SME product analytical results and the Mn from the glass analysis.[15]

In the second attempt, dried SME product was divided into four nearly equal portions. The first portion was heated to 1150 °C and held approximately fifteen minutes. The remaining portions were then added incrementally, allowing the crucible to return to temperature between each addition, resulting in a total time at 1150 °C of four hours. The sample was then quickly quenched to ambient temperature by placing the crucible in a shallow pan of water. No water contacted the glass during cooling. This glass was fabricated without incident. It appeared black and shiny, with no visible salt layer, crystals, or other inhomogeneities. This glass is referred to as the SB6 Qualification Glass and was used for the glass chemical and PCT analyses.

2.4.2 *Glass Dissolution Methods and Analyses*

To support compositional analysis, a portion of the SB6 Qualification Glass had to be dissolved. In order to enhance dissolution, approximately 4 g of the glass was crushed and ground using agate cups, balls and caps in a mechanical pulverizing mixer mill. The glass was sieved and only the portion that passed through a 200 mesh (<75 μm) brass sieve was used for the dissolutions. Weighed amounts (nominally 0.25 g) of the crushed glass were then dissolved remotely by two different methods to ensure that all the elements of interest were dissolved and could be analyzed. The two methods were a sodium peroxide fusion (PF) at 675 °C followed by a HNO_3 uptake, and a mixed acid dissolution (MA) in sealed vessels at 115 °C using a combination of HF, HCl, and HNO_3 acids. Boric acid was added to this latter dissolution method to complex excess fluoride. The solutions of the dissolved glass were diluted to known volumes so that approximately 15 mL aliquots could be safely removed from the Shielded Cells without exposing personnel to excess radiation.

The aliquots were then submitted to AD where they were analyzed by ICP-AES. Aliquots of the peroxide fusion dissolutions were also submitted for ICP-MS analysis and radioactive counting techniques. Concurrent with each set of dissolutions in the Shielded Cells, three samples of the ARG-1 and the Low Activity Test Reference Material (LRM) were also dissolved to determine if the dissolutions were complete and the resulting analyses accurate. With each set of samples sent to AD, two samples of a multi-element standard containing known concentrations of Al, B, Fe, Li, Na, and Si were also submitted.

2.4.3 *Standard ASTM 1285 Leach Test Procedure*

The durability of the SB6 Qualification Glass was measured using the ASTM 1285 standard nuclear waste glass leach test using the procedure prescribed in Test Method A.[16] This test is commonly referred to as the PCT. The purpose of the PCT is to confirm that the SB6 Qualification Glass has a durability that meets the criterion specified by the WAPS for repository acceptance.[17] WAPS 1.3 specifies that the mean concentrations of B, Li, and Na in the leachate, after normalizing for the concentrations in the glass, shall each be less than those of the Environmental Assessment (EA) glass.[18] These normalized concentrations represent the concentration of leached glass in PCT assuming all elements in the glass are soluble. DWPF complies with this criterion by demonstrating that the mean PCT results are at least two standard deviations below the mean PCT results of the EA glass.

The ASTM 1285 Test Method A is a crushed glass (-100 to +200 mesh or 75 to 149 μm) leach test at 90 °C for 7 days using DI water in sealed stainless steel vessels. The test was performed in quadruplicate for the SB6 Qualification Glass. Duplicate blanks and triplicate samples of the standard glass [Accepted Reference Material (ARM)] and triplicate samples of the EA glass were also tested with the samples. In the PCT, 10 mL of DI water are used for each gram of glass. Nominally 1.7 g of glass and 17 mL of DI water were used in stainless steel vessels that were sealed tightly and weighed. After 7 days at 90 °C, the containers were removed from the oven, allowed to cool, weighed to determine water loss, and then opened. Due to the radioactivity of the glass, the initial portion of the test was performed remotely in a Shielded Cell using manipulators. The leachates from each vessel were then decanted into a clean scintillation vial. The radioactivities of the leachates were low enough so they could be transported to a radiochemical hood where they could be handled directly. The pH of each leachate was measured and then filtered through a 0.45 μm filter and acidified to 1 volume percent HNO_3. The leachates were then diluted and submitted to AD, where the concentrations of B, Na, Li, and Si, were determined using ICP-AES.

2.4.4 *Glass Density Measurement*

The density of the SB6 Qualification Glass was measured using a pycnometry technique remotely in the Shielded Cells. A graduated cylinder was calibrated at multiple volume marks for measured weight of DI water. This same graduated cylinder was used for the density measurements. Small pieces of glass were added to the empty cylinder and the total dry mass was recorded. Incremental additions of water were added to the cylinder with the glass, total volume noted, and total mass recorded after each addition of water. Three water additions were recorded and used to calculate the density of the glass.

3.0 Results and Discussion

This section is divided into three major parts: 1) sludge washing, 2) DWPF Chemical Process Cell Simulation, and 3) glass fabrication and durability testing.

3.1 Sludge Washing

The SRNL Tank 51 SB6 qualification sample was washed per Tank Farm plans of November 10, 2009[9]. A mass balance showing wash, decant, and sample amounts is given in Appendix B.

The as-received and final washed sludge was characterized (elemental analyses, weight % solids, supernate analyses). Densities, anions, and elementals of each decant were measured. More extensive characterization (weight % solids, aqua regia digestions of slurry) was completed after Decants C and F to ensure washing was progressing as planned (e.g., no excessive dissolving or precipitation of solids, sulfur removal from solids). Decant C was chosen because a Pu stream was added during Washes B and C. Decant F was chosen because Wash F included a sodium nitrite addition for corrosion control.

Key analytical results (density, weight percent solids, selected supernate constituents, and major elementals in total solids) are presented in Table 3-1 and Table 3-2. Additional results can be found in Appendix C. Washing (decant) results are presented graphically in Figure 3-1.

The results show that washing proceeded as expected. Soluble species concentrations decreased (both in the supernate and in the total solids), and insoluble species concentrations increased (in the total solids). It should be noted that the mercury result from Post Decant C is lower than expected based on Fe to Hg ratios. Both elements are primarily insoluble in the caustic sludge. Therefore the ratio should be nearly content. However, Fe to Hg for wash C is less than 2.5 as compared to over 3 for the other analyses.

The results also show that aluminum is partially soluble. During washing, the aluminum in the supernate decreased (similar to sodium), but its concentration in the total solids initially decreased, but then increased after Decant C.

In comparing slopes in Figure 3-1, it appears that the slope for sulfur and sulfate is less than the slopes for sodium, nitrite, nitrate, and aluminum (from As-Received to Decant C). This suggests that a portion of undissolved sulfur is dissolving during washing.

During washing, about 15% of the total S as an undissolved species was observed (see Figure 3-2). Note that "undissolved" does not imply that this S cannot dissolve during washing operations; it just indicates that it was not soluble under the current sludge conditions. It can also be seen in Figure 3-2 that there is a small, but consistent difference between the soluble S from ICP-AES and the soluble sulfate by IC, expressed in the figure on a molar basis, for the SB6 Qualification sample during washing. This difference averaged 17.5 % across the eight washes, but the as-received material did not show any difference between soluble S and sulfate. Hence the difference in soluble S and sulfate seen during sludge batch washing appears to be due to the contribution of S from the HM sludge rather than the PUREX sludge.

Table 3-1. Major Elements in the Tank 51 SB6 Qualification Sample†

	As-Received	Post Decant C	Post Decant F	Post Decant I (SB6 SRAT Receipt)
Element	**Wt % of Total Solids**			
Al	6.01	5.98	7.81	11.0
Ca	0.140	0.242	0.357	0.527
Fe	2.87	4.80	7.76	11.8
Hg	0.872	2.05	2.06	3.12
K	0.117	<0.1	<0.1	<0.1
Mg	<0.1	<0.1	0.144	0.214
Mn	1.02	1.75	2.79	4.15
Na	30.2	29.5	24.9	15.4
Ni	0.415	0.677	1.12	1.69
P	<0.1	<0.1	0.118	0.143
S	1.04	0.785	0.663	0.375
Si	0.178	NM	NM	0.711
Th	0.731	NM	NM	2.98
U	0.601	1.11	1.73	2.46
Zr	<0.1	<0.1	0.129	0.156

NM = not measured. Only aqua regia digestions were performed after Decants C and F. Aqua regia is not an appropriate digestion for Si. Also, the digestions were not submitted for ICP-MS analysis, the method for Th quantification.

† Major elements are those detected at a concentration of greater than 0.1 wt % of total solids.

Table 3-2. Weight Percent Solids and Selected Supernate Constituents Measured During SB6 Washing

Parameter	As-Rcvd	Decant B	Decant C	Decant D	Decant E	Decant F	Decant G	Decant H	Decant I
Supernate Density (kg/L)	1.36	1.26	1.20	1.15	1.12	1.11	1.08	1.06	1.06
Slurry Density (kg/L)	1.38	NM	1.27	NM	NM	1.17	NM	NM	1.13
Total Solids (wt% of slurry)	38.01	NM	26.51	NM	NM	17.57	NM	NM	15.12
Dissolved Solids (wt% of supernatant)	34.03	NM	21.56	NM	NM	11.29	NM	NM	5.80
Insoluble Solids (wt% of slurry)	6.04	NM	6.31	NM	NM	7.08	NM	NM	9.89
Soluble Solids (wt% of slurry)	31.98	NM	20.20	NM	NM	10.49	NM	NM	5.23
Calcined Solids (wt% of slurry)	25.60	NM	NM	NM	NM	13.22	NM	NM	11.89
Sodium (M)	8.55	6.09	4.41	3.25	2.45	2.14	1.62	1.28	1.08
Nitrite (M)	1.27	0.752	0.498	0.440	0.262	0.414	0.392	0.297	0.237
Nitrate (M)	1.27	0.756	0.493	0.449	0.285	0.213	0.236	0.144	0.124
Sulfate (M) †	0.184	0.0980	0.0676	0.0485	0.0421	0.0290	0.0219	0.0174	0.015
Sulfur (M) †	0.181	0.110	0.073	0.058	0.049	0.037	0.0272	0.0174	0.018
Aluminum (M)	0.798	0.485	0.355	0.258	0.190	0.153	0.117	0.091	0.075

†Sulfate was determined from IC analysis, while sulfur was determined from ICP-AES analysis.

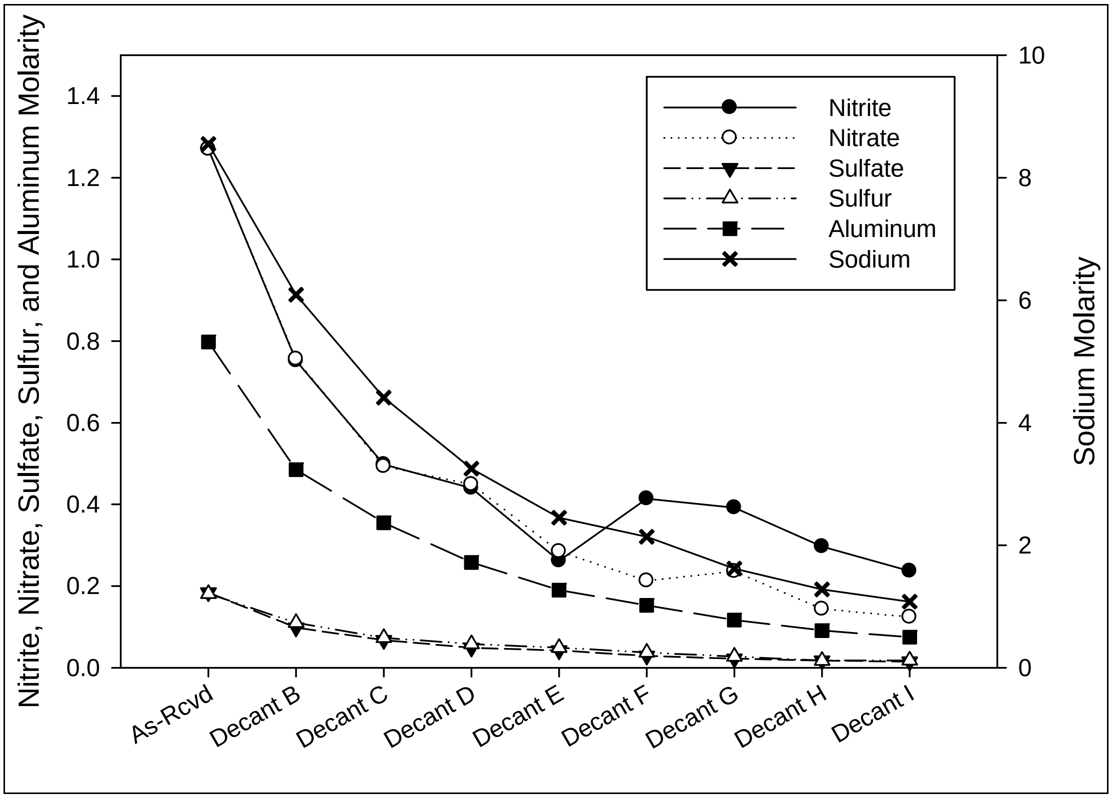


Figure 3-1. Graphical Presentation of Major Supernate Constituents

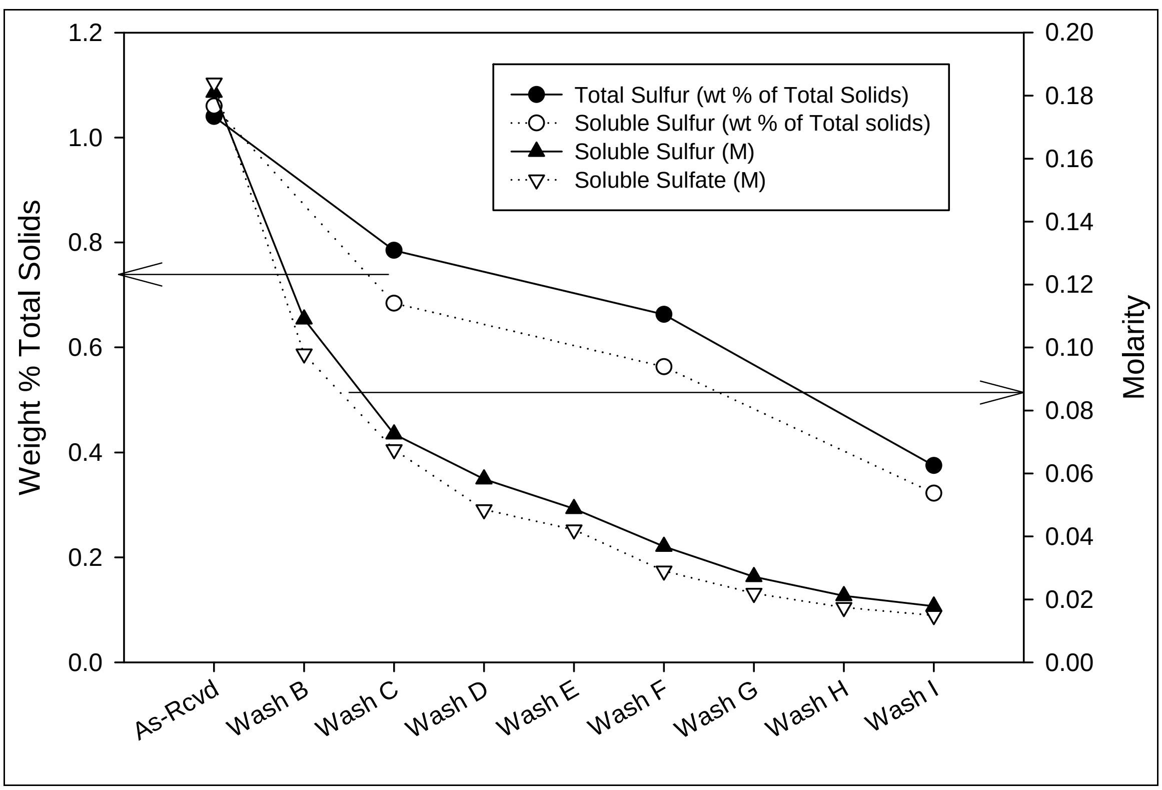


Figure 3-2. Graphical Presentation of Sulfur and Sulfate During Washing

Presented in Table 3-3 are the measured noble metals and other elements specifically requested in the TTR in the washed Tank 51 SB6 (SRAT receipt) solids. Noble metals catalyze hydrogen generation in the SRAT and SME cycles, giving qualitative guidance on SRAT acid stoichiometry.

Table 3-3. SRAT Receipt Tank 51 SB6 Qualification Sample Noble Metals and Minor Elements

Element	Wt % of Total Solids	Element	Wt % of Total Solids
Ru	0.0924	Cr	0.0448
Rh	0.0187	Co	0.00869
Pd	0.00304	Cu	0.0711
Ag	0.0138	Pb	0.0158
As	<0.0010	Sb	<0.014
Ba	0.0917	Se	<0.0021
Be	<0.00074	Ti	0.0164
Cd	0.00799		

Anions, total inorganic carbon, and total base are required for SRAT acid calculations. These results are presented in Table 3-4 and Table 3-5.

Table 3-4. SRAT Receipt Tank 51 SB6 Qualification Sample Anions and Carbon

Analyte	mg/kg slurry
Bromide	<100
Chloride	<100
Fluoride	<100
Formate	100
Nitrate	6,840
Nitrite	10,000
Oxalate	<100
Phosphate	<100
Sulfate	1200
Total Inorganic Carbon	913
Total Organic Carbon	483
Total Carbon	1400

Table 3-5. SRAT Receipt Tank 51 SB6 Qualification Sample Total Base, Free Hydroxide, and Other Bases Excluding Carbonate

Analyte	Mol/L Slurry
Total Base	0.58
Free OH^-	0.40
Other Base Excluding CO_3^{2-}	0.12

Slurry samples were prepared and submitted for VOA and SVOA analysis. No VOA or SVOA analytes were detected.

Sludge level was recorded during settling. These results are presented graphically in Figure 3-3. Observations (time, sludge level) are shown in Appendix E. The observations have been normalized (observed sludge level/starting sludge level for each wash) for comparison. It should be noted that Decant A, from the as-received Tank 51 sample was a relatively small decant. Sludge from Tank 4 (already present in the SRNL sample), not wash water, was added to Tank 51 prior to this decant. Thus, settling was not expected to be similar to the other washes. See Appendix A for the Tank Farm washing strategy which SRNL simulated. It appears that settling rate increased during washing up to Decant D. Settling during the remaining washes was comparable.

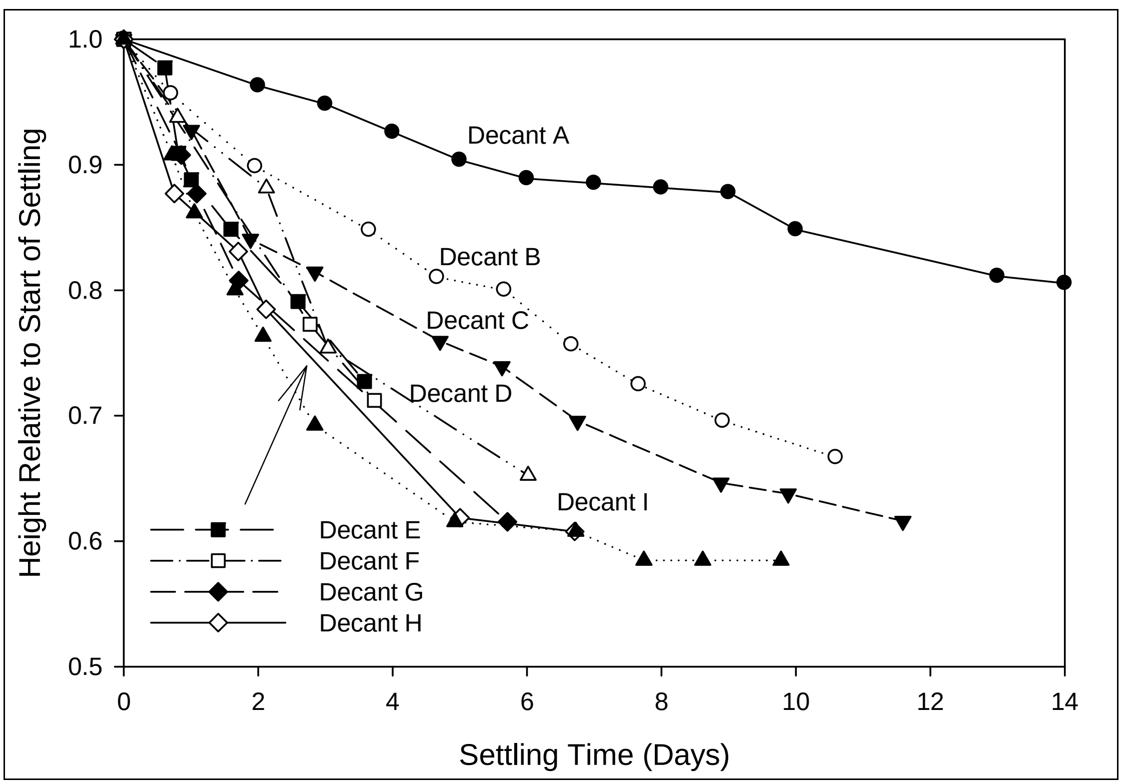


Figure 3-3. Setting During Washing.

3.2 Chemical Process Cell Simulation

3.2.1 *SRAT Cycle Results and Discussion*

Following washing of the Tank 51 SB6 qualification sample, the material was used in DWPF CPC simulations. The initial step of the simulations is the acid calculation to estimate the required acid necessary to complete reactions. This calculation uses measured analytical inputs. Errors in these measurements can result in too little acid being added resulting in incomplete reactions or too much acid being added resulting in excess formic acid causing high hydrogen generation rates. Analytical results of the SRAT receipt sample are given in Section 3.1. All inputs for the acid calculation are presented in Table 3-6. The non measured inputs (for example, acid stoichiometric factor, formic acid destruction, etc.) were based on simulant tests.[2] Note that characterization results of the SRAT receipt sample are presented in Section 3.1.

Table 3-6. Acid Calculation Inputs for the Tank 51 SB6 Qualification CPC Simulation

Measurement/Assumption	Units	Result
Total Solids	wt% of slurry	15.12
Insoluble Solids	wt% of slurry	9.89
Soluble Solids	wt% of slurry	5.23
Calcined Solids	wt% of slurry	11.89
Slurry Density	g/mL slurry	1.13
Supernatant Density	g/mL supernatant	1.06
Hg	wt% of total solids	3.12
Mn	wt% of calcined solids	5.28
Nitrite	mg/kg slurry	10,000
Nitrate	mg/kg slurry	6,840
Formate	mg/kg slurry	100
TIC	mg/kg slurry	913
Total Base	mol/L slurry to pH = 7	0.582
Conversion of Nitrite to Nitrate in SRAT Cycle	gmol NO_3^-/100 gmol NO_2^-	-10
Destruction of Nitrite in SRAT and SME cycle	% of starting nitrite	100
Destruction of Formic acid charged in SRAT	% of total formate	32
Percent Acid in Excess of Stoichiometric Ratio	%	115
SRAT Product Target Total Solids	wt% of SRAT Product	25
Predicted and/or Target REDOX	Fe^{+2} / ΣFe	0.2
No. of basis antifoam additions added during SRAT cycle	N/A	10
Destruction of Formic acid in SME	% of SRAT Product formate	5
Destruction of Nitrate in SME	% of SRAT Product nitrate	5
Assumed SME density	g/mL slurry	1.25
No. of basis antifoam additions added during SME cycle	N/A	10
Sludge Oxide Contribution in SME (Waste Loading)	% sludge oxides	34
SME Product Target Total Solids	wt% of SME Product	45

The primary results of the acid calculation (the acid requirements) are presented in Table 3-7. Note that DWPF reviewed the acid calculation.

Table 3-7. SRAT Cycle Acid Requirements

Parameter	Value
Calculated Stoichiometric Acid (100% stoichiometry), moles/L	1.12
Actual Acid to Add (stoichiometric amount x % excess acid), moles/L	1.29
Ratio of Formic Acid to Total Acid	0.89

The SRNL Tank 51 SB6 SRAT cycle (designated as SC-9 SRAT Cycle) began on February 22, 2010. The cycle began by concentrating the SRAT receipt material from 9.9 wt% insoluble solids to a target of 12 wt% insoluble solids by boiling and removing water. Foaming was constant during caustic boiling. 700 ppm of antifoam was added during the five hours of boiling/concentration to keep the foam from reaching the top of the vessel. Based on a mass balance calculation, the SRAT material was concentrated to 12.7 wt% insoluble solids. At the completion of caustic boiling, acid addition began. Nitric acid was completed without incident, but major foaming occurred approximately two thirds of the way into formic acid addition. Because of the loss of material in the foaming incident, the cycle was suspended, the remaining material was removed and the mass determined, the remaining amount of formic acid to add was recalculated, and a new SRAT/SME apparatus was installed.

The formic acid addition for the SRAT cycle resumed with the remaining material and the new apparatus on March 3, 2010. The SRAT product was concentrated to a target of 25 wt% total solids, and the contents were boiled at reflux for 35 hours at a DWPF-scaled bolup rate of 2,500 lb/h steam (see below for a discussion of Hg removal and boilup rate). Foaming was not observed during the conclusion of formic acid addition. Foaming was observed, but was not excessive during boiling. Antifoam addition times and amounts after the completion of acid addition are given in Table 3-9.

Table 3-8. Antifoam Additions During Caustic Boiling/Concentration

Time Relative to Start of Boiling (h:mm)	Antifoam Addition (ppm)
0:00	200
0:15	100
0:35	100
1:50	100
2:15	100
3:00	100
4:45	Dewater Complete

Table 3-9. Antifoam Additions After Formic Acid Addition

Time Relative to Completion of Formic Acid Addition (h)	Antifoam Addition (ppm)
0	600
8	100
12.5	100
13.5	100
21.5	100
28.5	100

Following the SRAT Cycle, an analytical sample was pulled, and the sample was characterized with results given in Table 3-10. Results show that nitrite was destroyed to less than 1,000 mg/kg, but mercury was not removed to the DWPF target of 0.6 wt% of the total solids.

After re-checking the SRNL acid calculations, it was discovered that the boilup rate was scaled to 2,500 lb/h steam, which had been used in a previous radioactive run to simulate DWPF's lower boil-up rate, instead of the design basis of 5,000 lb/h. Therefore, prior to the SME cycle, the sludge was boiled under reflux to remove additional mercury (nine hours at a scaled boilup rate of 5,000 lb/h).

Based on boiling at half the design boilup rate, the mercury would be expected to be approximately 1.5 wt% of the total solids. The measured result was lower – 1.1 wt % of the total solids, yielding s steam stripping rate of slightly more than the predicted 750 g steam/g Hg. Additional reflux time, at the DWPF rate of 5,000 lb/h, was calculated to reach the target of 0.6 wt% of total solids (750 g steam/g mercury). Following the reflux time (prior to the SME cycle), a sample was pulled. The result was 0.59 wt% mercury in the total solids. These results are consistent with expectations – mercury removal rate decreases slightly as mercury is removed. These results also show that 750 g of steam per g of mercury is adequate for mercury removal. Note that the vessel was purged at a scaled SME purge rate since the SME was begun immediately after this reflux time; the off-gas is presented in the SME cycle section, specifically Figure 3-6.

In addition to primary anions, SRNL analyzed the SRAT product for ammonium. SRNL also analyzed the SRAT dewater for ammonium. None was detected (<200 mg/kg).

Table 3-10. Characterization Results of the SB6 Qualification SRAT Product

Analysis	Result
Weight % Total Solids (slurry basis)	25.9
Weight% Dissolved Solids (supernate basis)	9.35
Weight % Insoluble Solids (slurry basis)	18.3
Weight% Soluble Solids (slurry basis)	7.6
Weight % Calcined Solids (slurry basis)	17.7
Slurry Density (g/mL)	1.23
Supernate Density (g/mL)	1.06
pH	10
Fluoride (mg/kg slurry)	<1,000
Formate (mg/kg slurry)	43,600
Chloride (mg/kg slurry)	<1,000
Nitrite (mg/kg slurry)	<1,000
Nitrate (mg/kg slurry)	26,200
Phosphate (mg/kg slurry)	<100
Sulfate (mg/kg slurry)	2,370
Oxalate (mg/kg slurry)	<100
Ammonium (mg/kg slurry)	<200
Total Carbon (mg/kg slurry) †	17,400
Total Inorganic Carbon (mg/kg slurry)	1,800
Total Organic Carbon (mg/kg slurry)	15,600
Mercury (wt % of total solids)	1.1

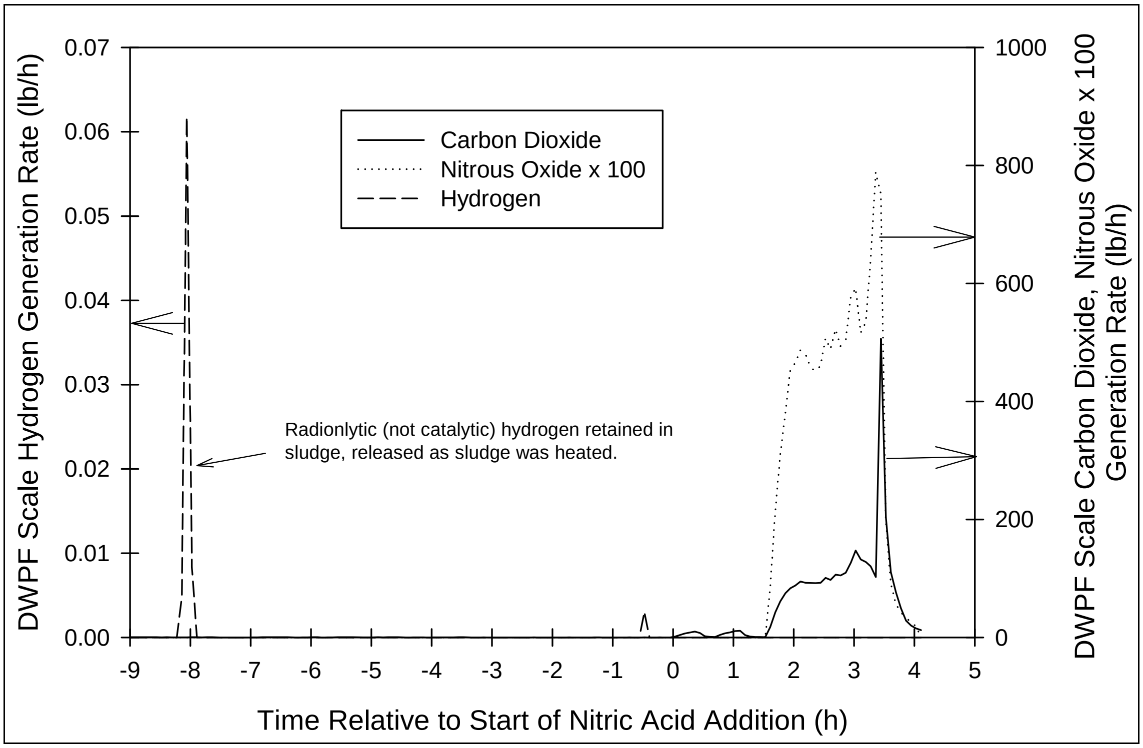
† Total Carbon = Total Inorganic Carbon + Total Organic Carbon

Off-gas data are presented in Table 3-11 (peak generation rates and concentrations), Figure 3-4 (off-gas during caustic boiling and acid addition up to the foam-over event), and Figure 3-5 (offgas during the completion of formic acid addition through the completion of the SRAT cycle). As expected, there was very little gas generation during boiling/concentration. Carbon dioxide and nitrous oxide generation began when nitric acid began. Peak carbon dioxide generation was observed just prior to the foam-over event. Carbon dioxide and nitrous oxide generation continued when the SRAT cycle (and formic acid addition) was resumed Several hours after nitrite destruction, as evidenced by the drop in nitrous oxide generation, hydrogen generation increased to a peak, and then slowly declined.

Table 3-11. SB6 Qualification SRAT Cycle Maximum Observed Hydrogen, Carbon Dioxide, and Nitrous Oxide DWPF Scale Concentrations and Generation Rates

Gas	Maximum Gas Concentration (vol%)	Maximum Gas Generation Rate (DWPF lb/h)
Hydrogen	0.75	0.55
Carbon Dioxide	23.4 †	506 †
Nitrous Oxide	1.00	17.2

† This generation rate and concentration was observed just prior to the foaming event. The peak carbon dioxide generation rate was increasing; thus, this value may not represent the maximum carbon dioxide generation rate.



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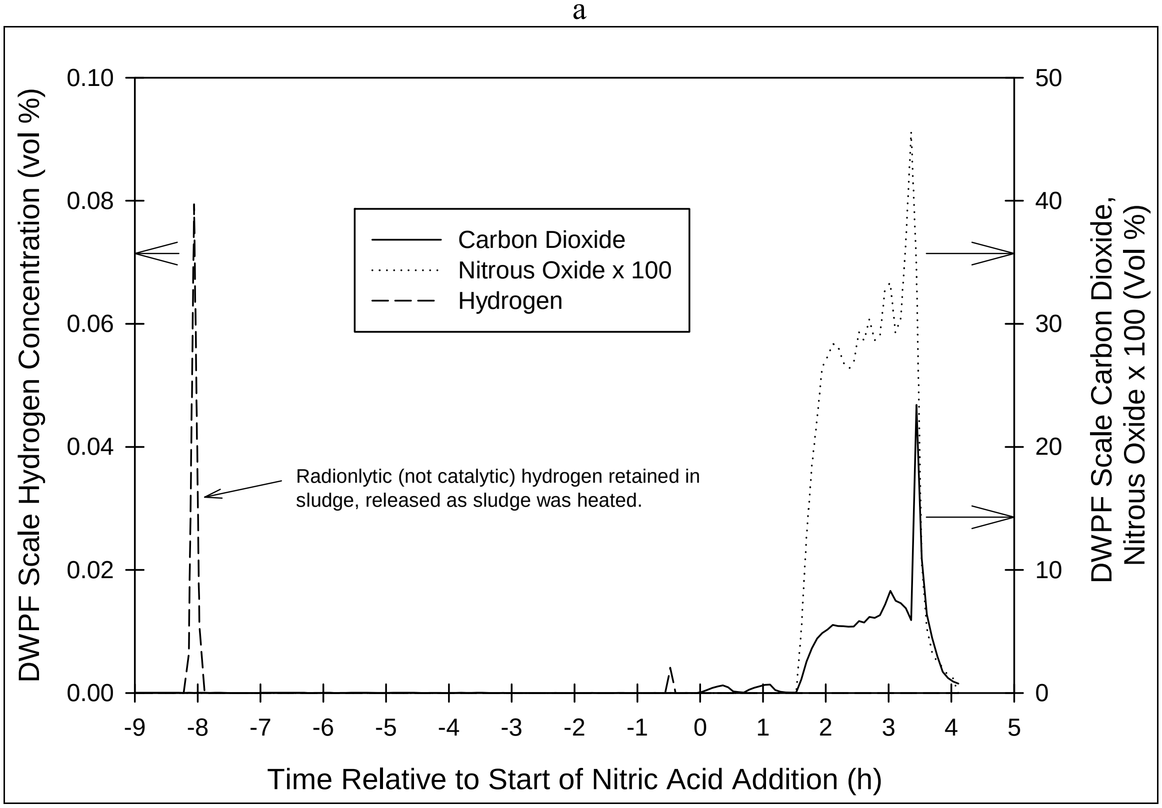

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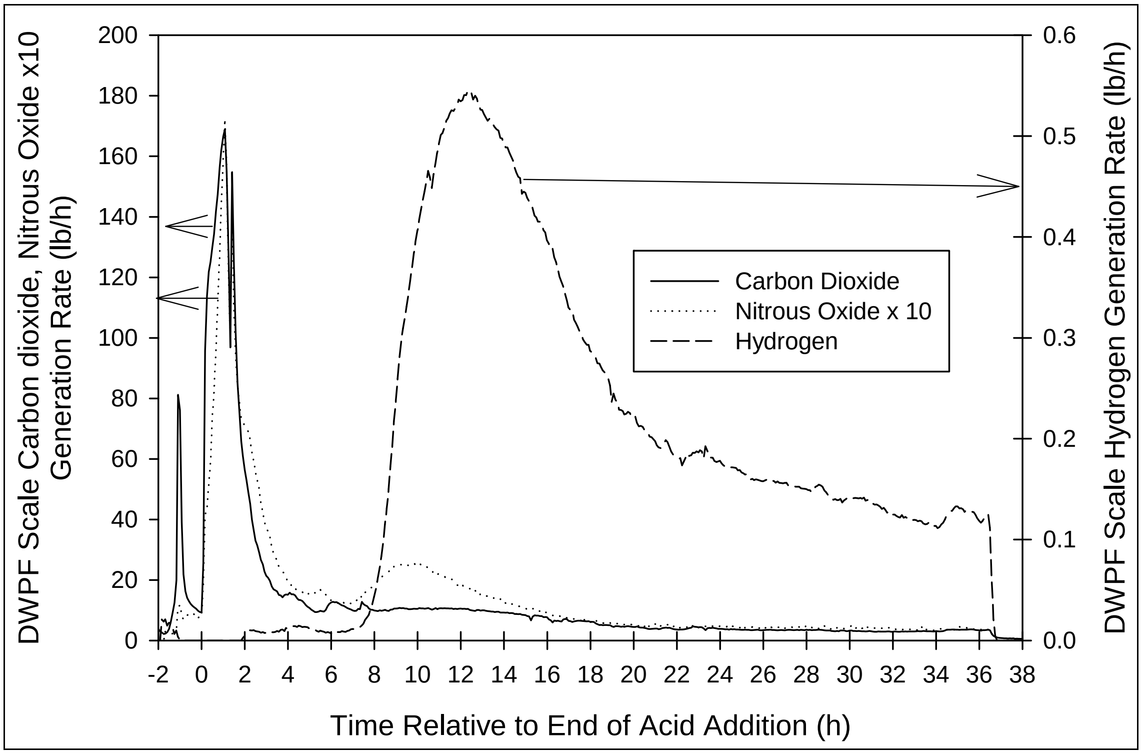
Figure 3-4. Plot of Gas Generation Rates (a) and Concentrations (b) During Caustic Boiling/Concentration and During Acid Addition Prior to Foaming Event



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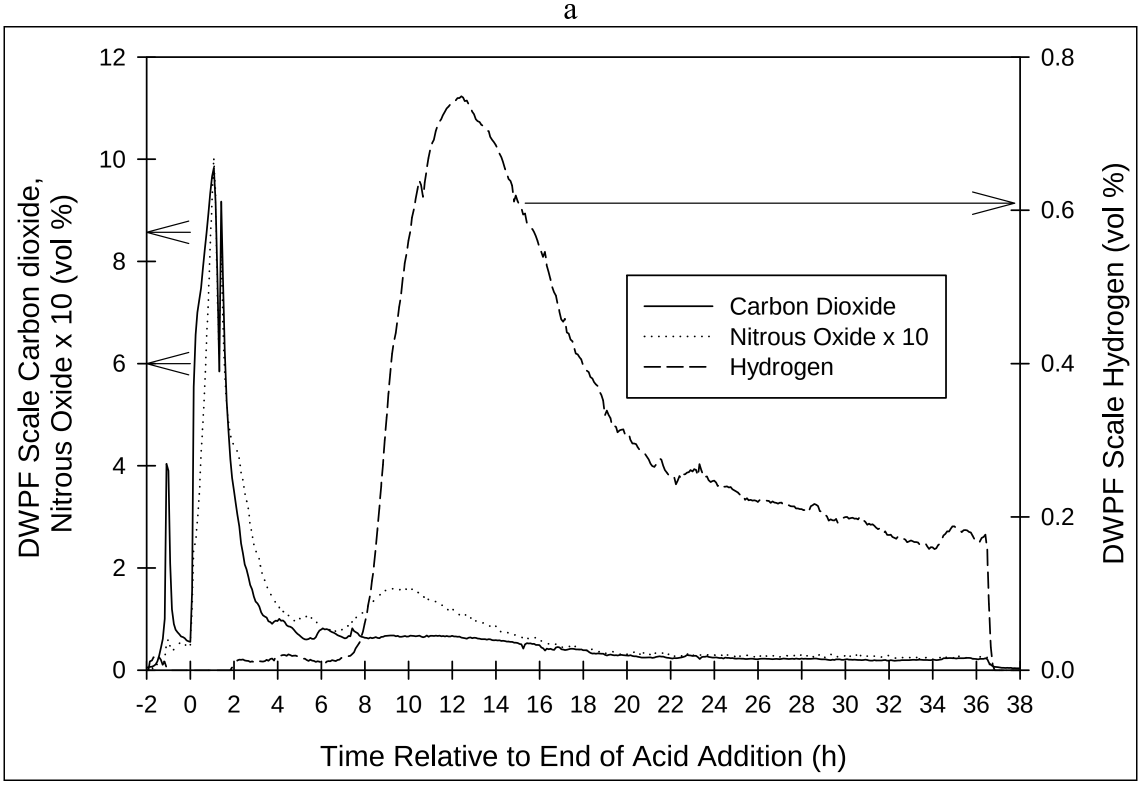

b

Figure 3-5. Plot of Gas Generation Rates (a) and Concentrations (b) Following Restart After Foam Over Event

3.2.2 *SME Cycle Results and Discussion*

The SRNL SB6 Qualification SME cycle began on March 18, 2010 with 470 g of SRAT product (based on mass balance calculations). Prior to the SME cycle, the SRAT product was boiled under reflux approximately nine hours to remove additional mercury. The SME cycle consisted of the addition and removal of water to simulate the addition and removal of decon water for five glass canisters (water was added and then boiled off five times). Frit 418 was then added in two equal batches (148 g total) along with an equivalent mass of water and formic acid (98.5 wt % water, 1.5 wt % formic acid). Frit addition was based on a target waste loading of 35 wt % calcined sludge. A sample of the SME product was vitrified, and the resulting glass was characterized and evaluated using the PCT.

The following observations were made during the SME cycle:

- An evaluation of mixing was not possible due to the small quantity of material. However, constant temperature during boiling and the absence of offgas spikes suggest that mixing was adequate.

- Excessive foaming was not observed during the SME cycle. 100 ppm antifoam was added prior to the SME cycle (during boiling under reflux to steam strip mercury). No additional antifoam was added throughout the SME cycle.

The SME product total solids was measured to be 52.5 wt %. The material was extremely thick and sticky; making sampling difficult. Therefore, condensate from the final SME dewater was added to the SME product to lower the total solids to 45 wt % (the targeted wt % total solids). At this lower total solids, sampling remained difficult. Because of limitations on the amount of SME product, its thickness, and its stickiness, supernate for dissolved solids and supernate density was not taken. Also, rheological measurements were not performed. The limited characterization data for the SME product at 52.5 wt % total solids (weight % total solids, weight percent calcined solids, and the calcine factor) is presented in Table 3-12.

Table 3-12. Physical Properties of As-Made SB6 SME Product (Prior to Dilution to 45 wt % Total Solids)

<table>
<tr><th>Property</th><th>Result</th></tr>
<tr><td>Wt % Total Solids</td><td>52.5</td></tr>
<tr><td>Wt % Calcined Solids</td><td>46.9</td></tr>
<tr><td>Calcine Factor (mass oxides/mass total solids)</td><td>0.89</td></tr>
<tr><td>Wt % Dissolved Solids</td><td rowspan="4">NA – SME product was too thick and sticky for adequate measurement.</td></tr>
<tr><td>Wt % Insoluble Solids</td></tr>
<tr><td>Wt % Soluble Solids</td></tr>
<tr><td>Supernate Density</td></tr>
</table>

Characterization results (density, anions, etc.) of the SME product after dilution with condensate to 45 wt % total solids are shown in Table 3-13. It is not surprising for total inorganic carbon to be detected in the SME product. The SRAT product had measurable total inorganic carbon (TIC) and was basic (pH=10). The pH of the SME product was not measured due to its thickness, but it would not be expected to be lower than the SRAT product.

Table 3-13. SB6 SME Product (at 45 wt % Total Solids) Density, Anion, and Cation Concentrations

Measurement	Result
Density, T = 21 °C (g/L)	1.42
Fluoride (mg/kg)	<50
Formate (mg/kg)	26,300
Chloride (mg/kg)	<50
Nitrite (mg/kg)	613
Bromide (mg/kg)	<50
Nitrate (mg/kg)	15,500
Phosphate (mg/kg)	<300
Sulfate (mg/kg)	1,450
Oxalate (mg/kg)	147
Ammonium (mg/kg)	<2,500
Total Inorganic Carbon (mg/kg)	1,800
Total Organic Carbon (mg/kg)	10,200
Total Carbon (mg/kg)	12,000 ‡

† Because of the stickiness of the sample and difficulty in removing it from the density measurement container, the density measurement was not repeated. It should be noted that the measured density is similar to what one would expect for a ~ 50 wt % total solids SME product.

‡ Total Carbon = Total Inorganic Carbon + Total Organic Carbon

Peak offgas generation and a plot of gas generation during the SME cycle are presented in Table 3-14 and Figure 3-6, respectively. It should be noted that the SRAT product was boiled under reflux prior to the start of the SME cycle; offgas generation during this reflux period is shown, but is not considered part of the SME cycle. Peak hydrogen generation (0.05 lb/h, DWPF scale) was below the DWPF limit of 0.223 lb/h. It is also noteworthy that hydrogen generation dropped drastically after the addition of decon water and never increased to the generation rate seen during the reflux time. The large vapor space in the SME vessel may have contributed to this.

Table 3-14. SB6 SME Cycle Maximum Observed Hydrogen, Carbon Dioxide, and Nitrous Oxide DWPF Scale Concentrations and Generation Rates

Gas	Maximum Gas Concentration (vol%)	Maximum Gas Generation Rate (DWPF lb/h)
Hydrogen	0.19	0.05
Carbon Dioxide	5.9	34
Nitrous Oxide	0.051	0.27

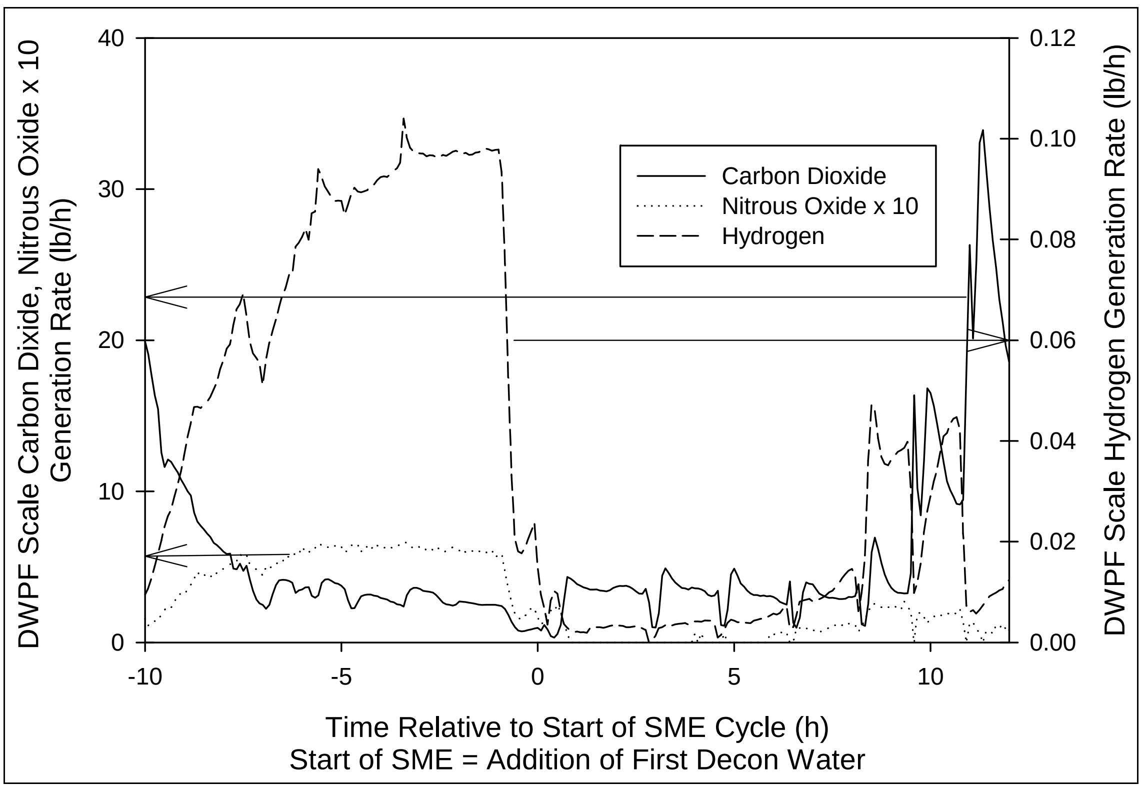

Figure 3-6. Offgas Data from the SB6 Qualification SME Cycle

3.2.3 *Rheology of CPC Materials*

Rheology results of the SRAT receipt and SRAT product material are presented in Table 3-15. As can be seen, concentration of the SRAT receipt material increased the yield stress to 12 Pa, higher than the design basis. This may be of concern in SB6 processing since rheology was not improved in the SRAT cycle. Therefore, SRNL does not recommend concentration prior to the SRAT cycle until further testing can be performed to define a more optimal endpoint.

Table 3-15. SB6 SRAT Receipt Slurry Rheology Data Before and After Concentration plus SRAT Product Rheology Data

Sample	Wt% Total Solids	Consistency (cP)	Yield Stress (Pa)
Washed Sludge Slurry (Design Basis) †	*13 – 19*	*4 – 12*	*2.5 – 10*
SRAT Receipt	15.1	8.9	8.3
SRAT Material Following Caustic Boiling/Concentration	19.7	12.3	12
SRAT Slurry (Design Basis) †	*18 – 25*	*5 – 12*	*1.5 – 5*
SRAT Product	25.9	15.2	21

† From *Basic Data Report: Defense Waste Processing Facility ludge Plant; Savannah River Plant 200-S Area*, DPSP-80-1033, Revision 10.

3.3 Glass Fabrication and PCT

Table 3-16 shows the elemental (excluding oxygen) composition of the SB6 Qualification Glass. Elements specifically requested in the TTR[1] (e.g., elements grater than 0.1 wt % in the sludge), along with elements necessary for PCCS calculations (e.g., Cu and Nd), are reported. Essentially all of the B, Li and Si and a portion of the Na are from the glass frit added to the SRAT product in order to prepare the glass. The frit used was Frit 418, which has a nominal composition of 76 wt % SiO_2, 8 wt % B_2O_3, 8 wt % Li_2O and 8 wt % Na_2O. Depending upon the element, the results in Table 3-16 represent an average of four or eight measurements resulting from the glass dissolution and analysis techniques mentioned in the experimental procedure. The dissolution and analytical technique used is noted in Table 3-16. next to each element. If one dissolution technique is listed then the result is an average of 4 replicates, but if both dissolution techniques are listed, the result for that element is an average of 8 replicates.

Table 3-16. Average of Elemental Concentrations Measured in SB6 Qualification Glass

Element	Wt %	% RSD	Digestion Method[a]	Detection Method
Ag	0.014	15.20	PF	ICP-MS
Al	4.528	4.44	MA/PF	ICP-AES
B	1.598	1.39	PF	ICP-AES
Ba	0.038	3.32	MA/PF	ICP-AES
Ca	0.275	2.77	MA	ICP-AES
Cd	0.003	4.87	MA	ICP-AES
Ce	0.049	2.60	PF	ICP-MS
Co	0.004	5.28	MA	ICP-AES
Cr	0.021	9.48	MA/PF	ICP-AES
Cu	0.025	9.88	MA/PF	ICP-AES
Fe	4.743	3.02	MA/PF	ICP-AES
Gd	0.025	8.08	MA/PF	ICP-AES
K	0.053	8.59	MA	ICP-AES
La	0.025	10.11	MA/PF	ICP-AES
Li	2.450	3.12	MA/PF	ICP-AES
Mg	0.098	3.85	MA/PF	ICP-AES
Mn	1.756	2.30	MA/PF	ICP-AES
Mo	0.007	5.23	MA	ICP-AES
Na	10.153	4.88	MA	ICP-AES
Nd	0.088	3.77	PF	ICP-MS
Ni	0.730	2.70	MA/PF	ICP-AES
P	0.052	4.33	MA	ICP-AES
Pb	0.007	5.81	MA	ICP-AES
S	0.087	13.41	MA	ICP-AES
Sb	< 0.011	N/A	MA	ICP-AES
Si	25.475	1.65	PF	ICP-AES
Sn	< 0.010	N/A	MA	ICP-AES
Sr	0.018	3.30	MA/PF	ICP-AES
Th	1.030	4.43	PF	ICP-MS
Ti	0.012	6.28	MA/PF	ICP-AES
U	0.874	3.40	PF	ICP-MS
V	< 0.004	N/A	MA	ICP-AES
Y	0.012	6.97	PF	ICP-MS
Zn	0.016	5.79	MA	ICP-AES
Zr	0.100	2.91	MA	ICP-AES
Sum	54.365			

[a] PF = Peroxide Fusion dissolution method, MA = Mixed Acid dissolution method

The measured SB6 Qualification Glass elemental concentrations reported in Table 3-16 were converted to an oxide basis and used to predict the properties of the glass based on the PCCS models and are listed in Table 3-17. All other elements listed in Table 3-16 and not found in Table 3-17 were ≤ 0.1 wt % oxide in the glass or were not used to complete the PCCS Measurement Acceptability Region (MAR) assessment. The predicted properties from this composition were then compared to SME acceptability criteria to evaluate whether this glass did indeed meet the DWPF processing and product quality constraints. Based on the measured composition, all of the predicted properties met the PCCS MAR criteria and a list of the predicted properties is found in Table 3-18.

Table 3-17. Measured SB6 Qualification Glass Composition on an Oxide Basis for Input into PCCS

Oxide	Weight %		Oxide	Weight %
Al_2O_3	8.555		Na_2O	13.686
B_2O_3	5.144		Nd_2O_3	0.103
BaO	0.042		NiO	0.929
CaO	0.385		P_2O_5	0.120
Ce_2O_3	0.057		PbO	0.008
Cr_2O_3	0.030		SO_4^{2-}	0.260
CuO	0.031		SiO_2	54.499
Fe_2O_3	6.780		ThO_2	1.172
K_2O	0.064		TiO_2	0.020
La_2O_3	0.030		U_3O_8	1.030
Li_2O	5.275		Y_2O_3	0.015
MgO	0.163		ZnO	0.019
MnO	2.268		ZrO_2	0.135
MoO_3	0.011		**SUM**	**100.830**

Table 3-18. PCCS Results for SB6 Qualification Glass

ΔG_p Value	-10.2055
NL[B (g/L)]	0.8868
NL[Li (g/L)]	0.8959
NL[Na (g/L)]	0.8789
T_L Prediction (ºC)	806.4878
Viscosity Prediction (P)	77.0212
Sum of Oxides (%)	100.5682 [a]
Nepheline Constraint Value	0.7102
Al_2O_3 (wt %)	8.5547
All PCCS MAR Criteria Met	yes

[a] Note that PCCS does not include SO_4 in its sum of oxides, accounting for the difference between the sum of oxides in this table and the sum in Table 6.

For the SB6 Qualification Glass, the waste loading was calculated based on Li_2O content and sum of oxides from the chemical analysis (from Table 3-17). The targeted Li_2O content of Frit 418 was also used (8.0 wt %). Using these results yields a waste loading of 34.4 wt %, which is nearly the same as the targeted 35 wt % WL.

Quadruplicate samples of the SB6 Qualification Glass were subjected to the PCT along with triplicate blanks, triplicate samples of the ARM and the EA reference glass as prescribed by the ASTM procedure. The results for the reference glasses and the blanks indicated that the test was acceptable. The water loss during the course of the test was very minimal and well within the acceptable amount of water loss as prescribed by the ASTM procedure. The blanks, and leachates from the ARM and EA references all had elemental and normalized releases within the accepted standard values. Results for the averaged normalized releases, based on B, Na, Li, Si, Al, Fe and U (grams of normalized element per liter of PCT leachate) are given in Table 3-19. The normalized releases for the SB6 Qualification Glass based on B, Na, and Li are more than an order of magnitude less than those for the EA glass. These releases are also predictable by the current durability models of the DWPF PCCS. A representation of predictability for ARM, EA and the SB6 Qualification Glass are in the plots for log normalized B, Li, Na and Si release as a function of del G_p as can be found Figure 3-7.

Table 3-19. PCT Results for ARM, EA and the SB6 Qualification Glass

Glass ID	NL (B) g/L	NL (Na) g/L	NL (Li) g/L	NL (Si) g/L	NL (Al) g/L	NL (Fe) g/L	NL (U) g/L
ARM[a]	0.46	0.50	0.56	0.27	0.19	N/A	N/A
EA[a]	17.42	13.65	9.91	4.01	<0.22	<0.02	N/A
SB6-QUAL[b]	0.63	0.90	0.81	0.46	0.51	0.27	0.57

[a] Average of 3 replicates
[b] Average of 4 replicates

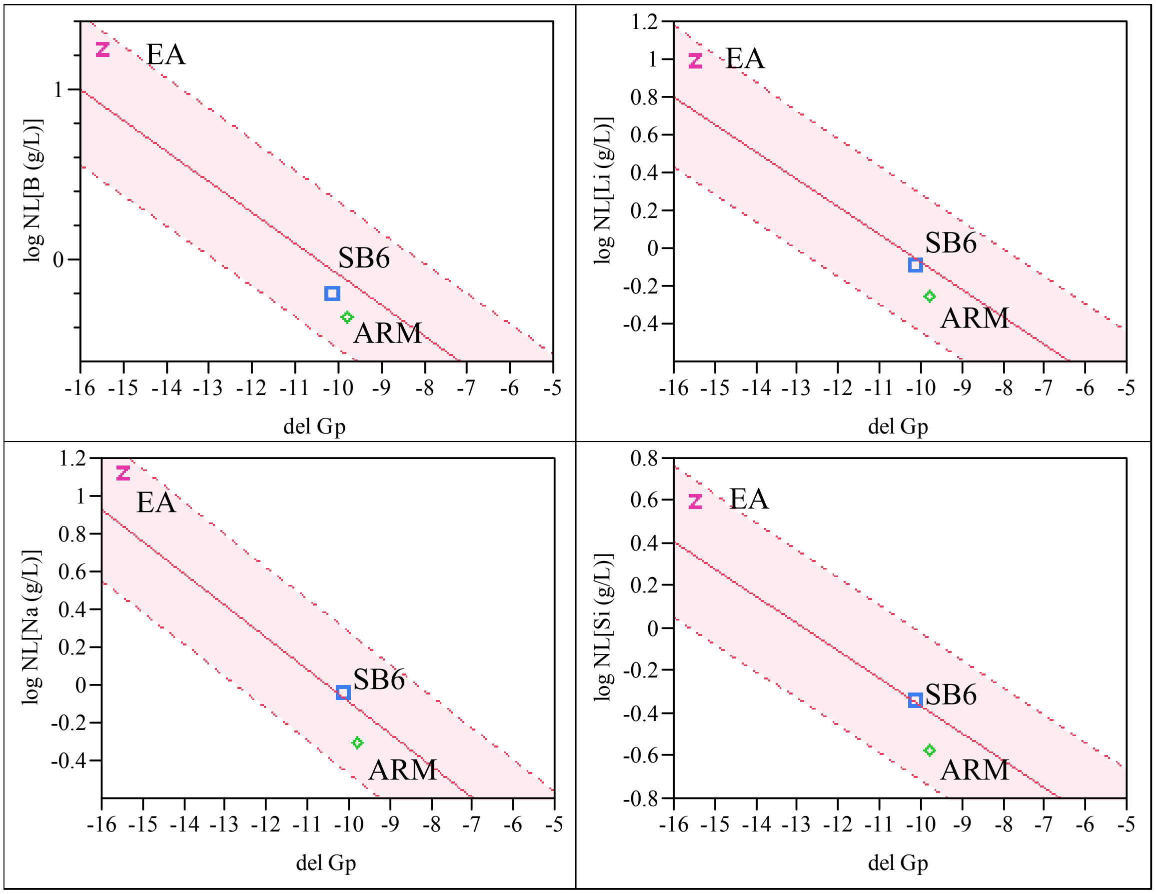

Figure 3-7. Fit of log Normalized Release of B, Li, Na and Si (g/L) vs. del G_p for the measured releases of ARM, EA and the SB6 Qualification glasses.

Density of the SB6 Qualification Glass was also measured using pycnometry. After measuring the glass with a calibrated graduated cylinder three times (the pycnometer utilized for these measurements), the average density was calculated to be 2.40 g/cm^3. This density is in the correct range for an alumino borosilicate glass.

4.0 Conclusions

The following conclusions are drawn from this demonstration

- Settling improved slightly as the sludge was washed.
- Thorium was detected in significant quantities (>0.1 wt % of total solids in the sludge). In past sludge batches, thorium has been determined by ICP-MS, seen in small quantities, and reported with the radionuclides. As a result of the high thorium, SRNL-AD has added thorium to their suite of ICP-AES elements.
- The acid stoichiometry for SRAT processing of 115%, or 1.3 mol acid per liter of SRAT receipt, was adequate to accomplish some of the goals of SRAT processing:

nitrite was destroyed to below 1,000 mg/kg (1,000 mg/kg nitrite was the method detection limit for this sample), and mercury was removed to below the DWPF target of 0.6 wt % of total solids (SRAT product was 0.59 wt %) with 750 g of steam per g of mercury. However, rheological properties did not improve and were above the design basis.

- Hydrogen generation rates did not exceed DWPF limits during the SRAT and SME cycles. However, the SRAT approached the limit.

	SRAT Cycle	SME Cycle
Peak DWPF Scale Generation Rate (lb/h)	0.55	0.05
DWPF Limit (lb/h)	0.65	0.223

- The glass fabricated with the Tank 51 SB6 SME product and Frit 418 was acceptable with respect to chemical durability as measured by the PCT. For example, the SB6 glass had a normalized boron release of 0.63 g/L, while the EA glass had a normalized release of 17.42 g/L. The PCT response was also predictable by the current durability models of the DWPF PCCS.

5.0 Recommendations and Path Forward

- In this demonstration, at the request of DWPF, SRNL caustic boiled the SRAT contents prior to acid addition to remove water (to increase solids concentration). During the nearly five hours of caustic boiling, foaming was constant; 700 ppm of antifoam was required to control foaming. SRNL recommends that DWPF not caustic boil/concentrate SRAT receipt prior to acid addition until further studies can be performed to provide a better foaming control strategy or a new antifoam is developed for caustic boiling.

- Based on this set of runs and a recently completed demonstration with the SB6 Waste Acceptance Product Specifications (WAPS) sample, it is recommended that DWPF not add formic acid at the design addition rate of two gallons per minute for this sludge batch. A longer acid addition time appears to be helpful in allowing slower reaction of formic acid with the sludge and possibly decreases the chance of a foam over during acid addition.

- Processing time (post acid addition concentration plus reflux) can be targeted from the mercury content. Mercury was stripped at the predicted 750 g steam per g Hg.

- As stated above, SRNL has completed CPC processing with a Tank 40 sample. SRNL did not concentrate prior to acid addition, and formic acid was added at a scaled one gallon per minute. A comparison of this run with the SB6 qualification run will be documented.

6.0 References

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Appendix A. Excerpt From November 10, 2009 Tank Farm Washing Spreadsheet

	A	BI	BJ	BK	BL	BM	BN	BO	BP	BQ	BR	BS
1		10/8/2009	11/07/09			11/12/09			11/19/09	12/12/09	12/14/09	
2	**Tank 51**	**Tank 51 after 4-51-1**	Decant A to Tank 4	Tank 51 after Decant	Add Caustic	Add IW	Pu Adds for SB6	Flush Volume at End of Pu Receipt for this Decant	Tank 51 after Additions	Decant B to 2F	Tank 51 after Decant B	Continuation of Pu Addsfor SB6
3	Initial tank Level (in)	**145.20**		**123.60**					**187.00**		135.10	
4	liquid volume (gal)	488552	75816	412736	**29000**	184609	5902	1500	633748	182169	451579	1967
5	sludge volume (gal)	21100		21100			23		22622		22622	8
6	settled sludge level (in)	119.10							129.10			
7	kg insol. solids	**159724**		159724			205		159928		159928	68
8	wt% insol solids	**6.00**		6.99					5.03		6.91	
9	decant level	123.10							133.10			
10	NO2/NO3											
11	additional nitrite solution		mole Na =						6.138	mole Na =		
12	additional volume	30727	2.45E+06			0				4.47E+06		
13	SpG	**1.3600**	1.360	1.360	1.530	1.000	1.140	1.033	1.260	1.260	1.2601	1.140
14	Na	**8.550**	8.550	8.550	19.125	0.021	3.035	1.200	6.481	6.481	6.4807	3.035
15	NO2	**1.2700**	1.270	1.270	0.000	0.011	0.002		0.830	0.830	0.8303	0.002
16	NO3	**1.2700**	1.270	1.270	0.000	0.000	1.677		0.847	0.847	0.8467	1.677
17	OH	**3.5300**	3.530	3.530	19.125	0.010	1.200	1.200	3.191	3.191	3.1910	1.200
18	Cl	0.0180	0.018	0.018	0.430	0.000	0.003		0.031	0.031	0.0314	0.003
19	SO4	**0.1840**	0.184	0.184	0.000	0.000	0.001		0.120	0.120	0.1198	0.001
20	F	0.0042	0.004	0.004	0.000	0.000	0.005		0.003	0.003	0.0028	0.005
21	CO3-2	**0.5030**	0.503	0.503	0.000	0.000	0.0002		0.328	0.328	0.3276	0.0002
22	AlO2	**0.7980**	0.798	0.798	0.000	0.000	0.017		0.520	0.520	0.5199	0.017
23	C2O4-2	0.0028	0.003	0.003	0.000	0.000	0.000		0.002	0.002	0.0018	0.000
24	PO4-3	**0.0065**	0.007	0.007	0.000	0.000	0.001		0.004	0.004	0.0043	0.001
25	K	**0.0188**	0.019	0.019	0.000	0.000	0.005		0.012	0.012	0.0123	0.005
26	Soluble Na2C2O4, M											
27	Insol Na2C204, kg	0										
28	**Heat Load**											
29	Sludge b/g	145212	0	145212	0.0	0.0	0	0	145212	0	145212	0
30	Sludge a	14178	0	14178	0.0	0.0	168	0	14346	0	14346	56
31	supernate b/g	11239	1744	9495	0.0	0.0	0	0	9495	2729	6765	0
32	supernate a	347	54	293	0.0	0.0	0	0	293	84	209	0
33	Temperature Basis, C	40							40			
34	C-LFL (corrected)	0.03734							0.03734			
35	NOeff (M)	1.8505							1.2337			
36	Rb/g	5.89							8.68			
37	Ra	35.01							45.35			
38	H2 b/g	0.92							1.34			
39	H2 alpha	0.51							0.66			
40	Total H2 (cuft/hr)	1.43							2.01			
41	Q(H2), Temp Corrected	1.64							2.30			
42	Vv, ft3 (for seismic)	115375							95774			
43	time for 25% LFL, instant release,											
44	time for 60% LFL, instant release,											
45	y0	0.007835							0.007835			
46	HLLCP	225.0							197.00			
47	Flammability Level	231.274							203.274			
48	Vv, ft3 (for vent loss)	75,012							88,142			
49	Q (for vent loss)	15.427							18.127			
50	Days to LFL (vent loss)	74.55							59.43			
51	Flammability Classification	slow							slow			
52	Q, seismic	23.73							19.70			
53	**Seismic Q-time, days**	83.50							46.57			
54	Mass TS, kg											
59	wt% TS	**38.00**										

	A	BT	BU	BV	BW	BX	BY	BZ	CA	CB	CC	CD
1			12/21/09	01/06/10	01/29/10		01/31/10	02/02/10	02/22/10		02/24/10	02/26/10
2	**Tank 51**	Additional Dilution to Continuation of Pu Adds	Add IW	Tank 51 after Pu Receipt & IW Add	Decant C to Tank 4	Tank 51 after Decant	Add IW	Tank 51 after IW Add	Decant D to 3H	Tank 51 after Decant	Add IW	Tank 51 after IW Add
3	Initial tank Level (in)			**187.00**		135.10		178.84		136.10		183.24
4	liquid volume (gal)	3500	176694	633740	182169	451571	153510	605081	**150000**	455081	170461	625542
5	sludge volume (gal)			22630		22630		22630		22630		17613
6	settled sludge level (in)			129.10				130.10				134.50
7	kg insol. solids			159997		159997		159997		159997		159997
8	wt% insol solids					7.31			~~252720~~	7.54		
9	decant level			133.10				134.10				138.50
10	NO2/NO3											0.99
11	additional nitrite solution		0		mole Na =		0		mole Na =		0	
12	additional volume		176694		3.20E+06		153510		1.97E+06		170461	
13	SpG	1.082	1.000	1.186	1.186	1.186	1.000	1.139	1.139	1.139	1.000	1.101
14	Na	2.000	0.021	4.644	4.644	4.644	0.021	3.471	3.471	3.471	0.021	2.531
15	NO2		0.011	0.595	0.595	0.595	0.011	0.447	0.447	0.447	0.011	0.3279
16	NO3	0.800	0.000	0.613	0.613	0.613	0.000	0.457	0.457	0.457	0.000	0.3328
17	OH		0.010	2.280	2.280	2.280	0.010	1.704	1.704	1.704	0.010	1.2426
18	Cl	1.200	0.000	0.029	0.029	0.029	0.000	0.022	0.022	0.022	0.000	0.0157
19	SO4		0.000	0.085	0.085	0.085	0.000	0.064	0.064	0.064	0.000	0.0464
20	F		0.000	0.002	0.002	0.002	0.000	0.001	0.001	0.001	0.000	0.0011
21	CO3-2		0.000	0.233	0.233	0.233	0.000	0.174	0.174	0.174	0.000	0.1267
22	AlO2		0.000	0.370	0.370	0.370	0.000	0.276	0.276	0.276	0.000	0.2012
23	C2O4-2		0.000	0.001	0.001	0.001	0.000	0.001	0.001	0.001	0.000	0.00071
24	PO4-3		0.000	0.003	0.003	0.003	0.000	0.002	0.002	0.002	0.000	0.0017
25	K		0.000	0.009	0.009	0.009	0.000	0.007	0.007	0.007	0.000	0.0048
26	Soluble Na2C2O4, M											
27	Insol Na2C204, kg											
28	**Heat Load**											
29	Sludge b/g	0	0.0	145212	0	145212	0.0	145212	0	145212	0.0	145212
30	Sludge a	0	0.0	14402	0	14402	0.0	14402	0	14402	0.0	14402
31	supernate b/g	0	0.0	6765	1945	4821	0.0	4821	1195	3626	0.0	3626
32	supernate a	0	0.0	209	60	149	0.0	149	37	112	0.0	112
33	Temperature Basis, C			40				40				40
34	C-LFL (corrected)			0.03734				0.03734				0.03734
35	NOeff (M)			0.8900				0.6649				0.4854
36	Rb/g			11.14				13.41				15.85
37	Ra			53.45				60.35				67.33
38	H2 b/g			1.69				2.01				2.36
39	H2 alpha			0.78				0.88				0.98
40	Total H2 (cuft/hr)			2.47				2.89				3.34
41	Q(H2), Temp Corrected			2.84				3.31				3.83
42	Vv, ft3 (for seismic)			95774				99602				97539
43	time for 25% LFL, instant release,											
44	time for 60% LFL, instant release,											
45	y0			0.007835				0.007835				0.007835
46	HLLCP			197.00				197.00				197.00
47	Flammability Level			203.274				203.274				203.274
48	Vv, ft3 (for vent loss)			88,142				88,142				88,142
49	Q (for vent loss)			18.127				18.127				18.127
50	Days to LFL (vent loss)			46.11				38.47				32.64
51	Flammability Classification			slow				slow				slow
52	Q, seismic			19.70				20.48				20.06
53	**Seismic Q-time, days**			36.48				31.73				25.86
54	Mass TS, kg											
59	wt% TS											

	A	CE	CF	CG	CH	CI	CJ	CK	CL	CM	CN	CO	CP
1		04/06/10		04/08/10	04/13/10	05/19/10		05/21/10	05/23/10	06/07/10		06/09/10	06/11/10
2	**Tank 51**	Decant E to 3H	Tank 51 after Decant	Add IW & Nitrite	Tank 51 after Nitrite & IW Add	Decant F to 3H	Tank 51 after Decant	Add IW	Tank 51 after IW Add	Decant G to 2F	Tank 51 after Decant	Add IW	Tank 51 after IW Add
3	Initial tank Level (in)		140.50		183.24		140.50		**187.00**		146.30		**187.00**
4	liquid volume (gal)	**150000**	475542	150000	625542	**150000**	475542	163215	638757	142857	495900	142857	638757
5	sludge volume (gal)		17613		17613		17613		17613		17613		17613
6	settled sludge level (in)				134.50				140.30				148.75
7	kg insol. solids		159997		159997		159997		159997		159997		159997
8	wt% insol solids	150930	7.47		5.86	150930	7.57				7.42		
9	decant level				138.50				144.30				152.75
10	NO2/NO3				**1.87**				1.89				
11	additional nitrite solution	mole Na =		18127		mole Na =		0		mole Na =			
12	additional volume	1.44E+06		131873		1.22E+06		163215		8.68E+05			
13	SpG	1.101	1.101	1.039	1.0861	1.086	1.086	1.000	1.0641	1.064	1.064	1.000	1.0498
14	Na	2.531	2.531	0.943	2.1503	2.150	2.150	0.021	1.6062	1.606	1.606	0.021	1.2517
15	NO2	0.328	0.328	0.934	0.4733	0.473	0.473	0.011	0.3552	0.355	0.355	0.011	0.2782
16	NO3	0.333	0.333	0.000	0.2530	0.253	0.253	0.000	0.1883	0.188	0.188	0.000	0.1462
17	OH	1.243	1.243	0.009	0.9468	0.947	0.947	0.010	0.7074	0.707	0.707	0.010	0.5514
18	Cl	0.016	0.016	0.000	0.0120	0.012	0.012	0.000	0.0089	0.009	0.009	0.000	0.0069
19	SO4	0.046	0.046	0.000	0.0352	0.035	0.035	0.000	0.0262	0.026	0.026	0.000	0.0204
20	F	0.001	0.001	0.000	0.0008	0.001	0.001	0.000	0.0006	0.001	0.001	0.000	0.0005
21	CO3-2	0.127	0.127	0.000	0.0963	0.096	0.096	0.000	0.0717	0.072	0.072	0.000	0.0557
22	AlO2	0.201	0.201	0.000	0.1529	0.153	0.153	0.000	0.1138	0.114	0.114	0.000	0.0884
23	C2O4-2	0.001	0.001	0.000	0.0005	0.001	0.001	0.000	0.0004	0.000	0.000	0.000	0.0003
24	PO4-3	0.002	0.002	0.000	0.0013	0.001	0.001	0.000	0.0009	0.001	0.001	0.000	0.0007
25	K	0.005	0.005	0.000	0.0036	0.004	0.004	0.000	0.0027	0.003	0.003	0.000	0.0021
26	Soluble Na2C2O4, M												
27	Insol Na2C204, kg										0		
28	**Heat Load**	[Na]/I.S. =	**28.474**			[Na]/I.S. =	**24.190**			[Na]/I.S. =	**18.843**		
29	Sludge b/g	0	145212	0.0	145212	0	145212	0.0	145212	0	145212	0.0	145212
30	Sludge a	0	14402	0.0	14402	0	14402	0.0	14402	0	14402	0.0	14402
31	supernate b/g	869	2756	0.0	2756	661	2095	0.0	2095	469	1627	0.0	1627
32	supernate a	27	85	0.0	85	20	65	0.0	65	14	50	0.0	50
33	Temperature Basis, C				40				40				40
34	C-LFL (corrected)				0.03734				0.03734				0.03734
35	NOeff (M)				0.4785				0.3578				0.2789
36	Rb/g				15.97				18.19				20.04
37	Ra				67.64				73.63				78.39
38	H2 b/g				2.36				2.68				2.94
39	H2 alpha				0.98				1.07				1.13
40	Total H2 (cuft/hr)				3.34				3.74				4.07
41	Q(H2), Temp Corrected				3.83				4.29				4.67
42	Vv, ft3 (for seismic)				97539				95774				95774
43	time for 25% LFL, instant release,												
44	time for 60% LFL, instant release,												
45	y0				0.007835				0.007835				0.007835
46	HLLCP				197.00				197.00				197.00
47	Flammability Level				203.274				203.274				203.274
48	Vv, ft3 (for vent loss)				88,142				88,142				88,142
49	Q (for vent loss)				18.127				18.127				18.127
50	Days to LFL (vent loss)				32.58				28.69				26.12
51	Flammability Classification				slow				slow				rapid
52	Q, seismic				20.06				19.70				19.70
53	**Seismic Q-time, days**				25.81				21.77				19.44
54	Mass TS, kg												
59	wt% TS												

	A	CQ	CR	CS	CT	CU	CV	CW	CX	CY	CZ	DA	DB
1		06/24/10		06/25/10	06/27/10	07/09/10		07/10/10	07/12/10	07/24/10		07/25/10	07/27/10
2	**Tank 51**	Decant H to 3H	Tank 51 after Decant	Add IW	Tank 51 after IW Add	Decant I to 2F	Tank 51 after Decant	Add IW	Tank 51 after IW Add	Decant J to 3H	Tank 51 after Decant	Add IW	Tank 51 after IW Add
3	Initial tank Level (in)		154.75		**187.00**		159.00		**187.00**		159.00		**187.00**
4	liquid volume (gal)	113198	525559	113198	638757	98280	540477	98280	638757	98280	540477	98280	638757
5	sludge volume (gal)		17613		17613		17613		17613		17613		17613
6	settled sludge level (in)				153.00				153.00				157.25
7	kg insol. solids		159997		159997		159997		159997		159997		159997
8	wt% insol solids	126360	7.12			224640	6.99			105300	7.03		
9	decant level				157.00				157.00				161.25
10	NO2/NO3				1.92				1.94				**1.96**
11	additional nitrite solution	mole Na =				mole Na =				mole Na =			
12	additional volume	5.36E+05				3.84E+05				3.27E+05			
13	SpG	1.050	1.050	1.000	1.0409	1.041	1.041	1.000	1.035	1.035	1.035	1.000	1.029
14	Na	1.252	1.252	0.021	1.0336	1.034	1.034	0.021	0.878	0.878	0.878	0.021	0.746
15	NO2	0.278	0.278	0.011	0.2308	0.231	0.231	0.011	0.1970	0.197	0.197	0.011	0.1684
16	NO3	0.146	0.146	0.000	0.1203	0.120	0.120	0.000	0.1018	0.102	0.102	0.000	0.0861
17	OH	0.551	0.551	0.010	0.4555	0.455	0.455	0.010	0.3869	0.387	0.387	0.010	0.3289
18	Cl	0.007	0.007	0.000	0.0057	0.006	0.006	0.000	0.0048	0.005	0.005	0.000	0.0041
19	SO4	0.020	0.020	0.000	0.0168	0.017	0.017	0.000	0.0142	0.014	0.014	0.000	0.0120
20	F	0.000	0.000	0.000	0.0004	0.000	0.000	0.000	0.0003	0.000	0.000	0.000	0.0003
21	CO3-2	0.056	0.056	0.000	0.0458	0.046	0.046	0.000	0.0388	0.039	0.039	0.000	0.0328
22	AlO2	0.088	0.088	0.000	0.0727	0.073	0.073	0.000	0.0615	0.062	0.062	0.000	0.0521
23	C2O4-2	0.000	0.000	0.000	0.0003	0.000	0.000	0.000	0.00022	0.000	0.000	0.000	0.00018
24	PO4-3	0.001	0.001	0.000	0.0006	0.001	0.001	0.000	0.0005	0.001	0.001	0.000	0.0004
25	K	0.002	0.002	0.000	0.0017	0.002	0.002	0.000	0.0015	0.001	0.001	0.000	0.0012
26	Soluble Na2C2O4, M								0.13503				0.17044
27	Insol Na2C204, kg		0				0		0		0		0
28	**Heat Load**	[Na]/I.S. =	**15.562**			[Na]/I.S. =	**13.215**			[Na]/I.S. =	**11.223**		
29	Sludge b/g	0	145212	0.0	145212	0	145212	0.0	145212	0	145212	0.0	145212
30	Sludge a	0	14402	0.0	14402	0	14402	0.0	14402	0	14402	0.0	14402
31	supernate b/g	288	1338	0.0	1338	206	1133	0.0	1133	174	958	0.0	958
32	supernate a	9	41	0.0	41	6	35	0.0	35	5	30	0.0	30
33	Temperature Basis, C				40				40				40
34	C-LFL (corrected)				0.03734				0.03734				0.03734
35	NOeff (M)				0.2305				0.1958				0.1665
36	Rb/g				21.41				22.56				23.67
37	Ra				81.83				84.61				87.24
38	H2 b/g				3.14				3.30				3.46
39	H2 alpha				1.18				1.22				1.26
40	Total H2 (cuft/hr)				4.32				4.52				4.72
41	Q(H2), Temp Corrected				4.95				5.19				5.41
42	Vv, ft3 (for seismic)				95774				95774				95774
43	time for 25% LFL, instant release,												
44	time for 60% LFL, instant release,												
45	y0				0.007835				0.007835				0.007835
46	HLLCP				197.00				197.00				197.00
47	Flammability Level				203.274				203.274				203.274
48	Vv, ft3 (for vent loss)				88,142				88,142				88,142
49	Q (for vent loss)				18.127				18.127				18.127
50	Days to LFL (vent loss)				24.50				23.30				22.25
51	Flammability Classification				rapid				rapid				rapid
52	Q, seismic				19.70				19.70				19.70
53	**Seismic Q-time, days**				17.95				16.83				15.85
54	Mass TS, kg												
59	wt% TS												

Appendix B. SRNL Sludge Batch 6 Washing Mass Balance

NOTE: The information presented in this appendix was published previously in memorandum SRNL-L3100-2010-00082, *Balance During SRNL Washing of Sludge Batch 6 Qualification Sample*.

Presented below is the mass balance during washing of the SRNL Sludge Batch 6 Qualification sample. Also included are an insoluble solids balance, volume observations throughout washing, and volumes of additions and removals (calculated from mass and density).

Please note the following:

- The mass of the as-received sample was measured. After that measurement, only the masses of additions and removals were measured.
- Weight percent insoluble solids were calculated from total solids and dissolved solids measurements.
- Volumes were calculated based on measured or estimated densities.
- Because volume is not conserved, a running volume balance is not presented. However, observed volumes and volumes of additions and removals (based on mass and density) are given.

The wt% insoluble solids after Decant I was determined to be 9.9 wt%. Based on the mass balance and the wt% insoluble solids, the total mass of insoluble solids after Decant I is calculated to be

9.9/100 x 2314 = 229 g,

or only 7% higher than the mass of insoluble solids (214 g) calculated from the mass balance. This suggests that there was no significant precipitation or dissolving of insoluble solids during washing at SRNL.

Explanation of Table Headings

Note that removals or subtractions are designated by placement in parentheses

Addition or ((Removal) – mass of material added or removed during a given washing step.

Running Total – sum of additions and removals.

Insoluble Solids (wt%) – the determined wt% insoluble solids at a given point during washing.

Insoluble Solids Added (Removed) – the mass of insoluble solids added or removed based on the sample size and the determined wt% insoluble solids.

Insoluble Solids Running Total – the sum of insoluble solids additions and removals.

Observed Total Volume - the total volume based on the graduations on the SRNL washing vessel. Note that observations were taken after each addition to track sludge settling.

Density of Add'n or Removal – the measured or estimated density of additions and removals. The density of the decants and sludge samples were measured. The densities of additions were estimated (e.g., the density of inhibited water was estimated to be 1.0 g /mL).

Calculated Volume Added (Removed) – the mass of an addition or removal divided by the density.

	Addition or (Removal) (g)	Running Total (g)	Insoluble Solids (wt%)	Insoluble Solids Added (Removed) (g)	Insoluble Solids Running Total (g)	Observed Total Volume (mL)	Density of Add'n or Removal (g/mL)	Calculated Volume Added (Removed) (mL)
Initial Sample	4,211.04	4,211	6.0	253	253	3,100	1.38	3,051
Subsample Removed	(530.16)	3,681	6.0	(32)	221	2,700	1.38	(384)
Decant A	(493.49)	3,187	-	-	221	-	1.36	(363)
Wash B Additions [a]	1,219.75	4,407	-	-	221	3,450	1.08	1,129
Decant B	(1,124.05)	3,283	-	-	221	-	1.26	(892)
Wash C Additions [a]	960.05	4,243	-	-	221	3,450	1.00	960
Decant C	(1,113.61)	3,130	-	-	221	-	1.20	(928)
Post Decant C Slurry Subsample	(55.40)	3,074	6.3	(3)	217	-	1.27	(44)
Wash D	776.28	3,850	-	-	217	3,200	1.00	776
Decant D	(843.82)	3,007	-	-	217	-	1.15	(734)
Wash E	862.74	3,869	-	-	217	3,300	1.00	863
Decant E	(839.25)	3,030	-	-	217	-	1.12	(749)
Wash F [b]	794.88	3,825	-	-	217	3,300	1.04	764
Decant F	(840.34)	2,985	-	-	217	-	1.11	(757)
Post Decant F Slurry Subsample	(53.03)	2,932	7.1	(4)	214	-	1.17	(45)
Wash G	791.25	3,723	-	-	214	3,250	1.00	791
Decant G	(736.68)	2,986	-	-	214	-	1.08	(682)
Wash H	695.24	3,681	-	-	214	3,250	1.00	695
Decant H	(579.21)	3,102	-	-	214	-	1.06	(546)
Wash I	550.93	3,653	-	-	214	3,250	1.00	551
Decant I Supernate Subsample [c]	(57.45)	3,596	-	-	214	-	1.06	(54)
Decant I	(1,281.29)	2,314	-	-	214	-	1.06	(1,209)
Post Decant I Sludge	-	*2,314*	*9.9*	-	*229 **	*2,050***	*1.13*	*NA*

* The Post Decant I slurry had an insoluble solids content of 9.9 wt% (calculated from measured total and dissolved solids). Based on this insoluble solids result, the calculated insoluble solids mass is 2,314 x 0.099 = 229 g.

** The final Post Decant I slurry volume is calculated to be 2314 g / 1.13 g/mL = 2,050 mL.

[a] Wash B and C additions included a Pu stream from H Canyon, neutralized to 1.2 M excess hydroxide, along with additional sodium hydroxide to simulate flushes from H Canyon.

[b] Wash F additions included significant sodium nitrite.

[c] A small sample of supernate was taken for analysis during Wash I settling. The results were used in the decision to stop washing at Wash I

Appendix C. SRNL Sludge Batch 6 Analytical Results

Presented in this appendix are more compete analytical results from sludge washing and the CPC simulations. These tables include additional analytes and relative standard deviations.

Table C - 1. Density and Weight Percent Solids for Tank 51 SB6 Qualification Sample

Property	Result	*%Rsd*
Slurry Density	1.38	*0.3*
Supernate Density	1.36	*0.4*
Wt% Total Solids (Slurry Basis)	38.0	*0.5*
Wt% Calcined Solids (Slurry Basis)	25.6	*0.6*
Wt% Dissolved Solids[a] (Supernate Basis)	34.0	*0.1*
Wt% Soluble Solids[b] (Slurry Basis)	32.0	*0.2* [c]
Wt% Insoluble Solids (Slurry Basis)	*6.0*	*2.5* [c]

[a] Also known as Uncorrected Soluble Solids
[b] Also known as Corrected Soluble Solids
[c] %RSD here is more correctly defined as % standard error for these calculated values
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 2. Concentrations of Elements in Total Dried Solids and Calcined Solids for the As-Received Tank 51 SB6 Qualification Sample

Element	Wt% Total Solids	Wt% Calcine Solids	%Rsd	Instrument	Prep Method
Ag	0.00288	0.00428	3.9	ICP-MS	AR
Al	6.01	8.93	1.2	ICP-AES	PF
B	0.0106	0.0158	5.4	ICP-AES	AR
Ba	0.0227	0.0337	2.3	ICP-AES	AR/PF
Be	0.000147	0.000219	2.8	ICP-AES	AR
Ca	0.140	0.207	2.1	ICP-AES	AR
Cd	0.00198	0.00294	8.8	ICP-MS	AR
Ce	0.0336	0.0499	0.8	ICP-MS	AR
Cr	0.0536	0.0795	6.0	ICP-AES	AR/PF
Cu	0.0174	0.0258	2.9	ICP-AES	AR/PF
Fe	2.87	4.26	2.5	ICP-AES	AR/PF
Gd	0.00724	0.0107	8.1	ICP-MS	AR
Hg	0.872	-	6.6	CVAA	AR
K	0.117	0.173	6.6	ICP-AES	AR
La	0.0170	0.0253	1.9	ICP-MS	AR
Li	0.00445	0.00661	2.5	ICP-AES	AR
Mg	0.0524	0.0778	2.4	ICP-AES	AR/PF
Mn	1.02	1.51	1.3	ICP-AES	AR/PF
Mo	0.00826	0.0123	7.3	ICP-AES	AR
Na	30.2	44.8	9.0	ICP-AES	AR
Nd	0.0627	0.0931	0.5	ICP-MS	AR
Ni	0.415	0.617	2.4	ICP-AES	AR/PF
P	0.0571†	0.0847	10	ICP-AES	AR
Pb	0.00434	0.00645	3.9	ICP-MS	AR
S	1.04	1.54	2.9	ICP-AES	AR
Sb	<0.0092	<0.014	-	ICP-AES	AR
Si	0.178	0.264	5.1	ICP-AES	PF
Sn	<0.0043	<0.0064	-	ICP-AES	AR
Sr	0.0106	0.0158	2.4	ICP-AES	AR/PF
Ti	0.00413	0.00613	9.4	ICP-AES	AR/PF
Th	0.731	1.08	2.4	ICP-MS	AR
U	0.601	0.892	1.1	ICP-MS	AR
V	<0.00059	<0.00088	-	ICP-AES	AR
Zn	0.0138	0.0204	11	ICP-AES	AR/PF
Zr	0.057	0.085	32	ICP-AES	AR

Results are Averages of Dissolution and Analysis of Four to Eight Aliquots of the SB6 Slurry.
† Reported result is the average of three replicates.
ICP-MS ≡ inductively coupled plasma – mass spectrometry, ICP-AES ≡ inductively coupled plasma – atomic emission spectroscopy, CVAA ≡ cold vapor atomic absorption spectroscopy, AR ≡ aqua regia digestion, PF ≡ peroxide fusion
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 3. Concentrations of Noble Metals and Silver in Total Dried Solids for the As-Received Tank 51 SB6 Qualification Sample

Element	Wt% Total Solids	%RSD
Ru	0.0227	0.7
Rh	0.00471	1.1
Pd	0.00134	1.9
Ag	0.00288	3.9

Results are Averages of Aqua Regia Digestions and ICP-MS Analyses of Four SB6 Slurry Aliquots .
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 4. Concentrations of Soluble Elements in the As-Received SB6 Qualification Sample Supernate

Element	Molarity	%RSD
Al	0.798	1.0
B	0.00557	0.2
Ca	0.000153*	5.4
Cr	0.00511	0.3
Fe	0.000105**	4.5
K	0.0188	3.4
Mo	0.000509	2.0
Na	8.55	1.0
P	0.00654	1.5
S	0.181	2.2
Zn	0.0000751	3.0

Results are Averages of Four SB6 Supernate Aliquots, diluted in acid, and analyzed by ICP-AES.
 * Result is the average of three measurements.
** Result is the average of two measurements.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 5. Concentrations of Anions on a Supernate Basis for the As-Received Tank 51 SB6 Qualification Sample

Anion	Molarity	%RSD
Br^-	<0.032	-
Cl^-	<0.073	-
F^-	<0.14	-
HCO_2^-	<0.057	-
NO_3^-	1.27	4.9
NO_2^-	1.27	4.7
CO_3^{2-}	0.503	1.2
$C_2O_4^{2-}$	<0.029	-
PO_4^{3-}	<0.027	-
SO_4^{2-}	0.184	10

Results are averages of four SB6 supernate aliquots diluted with water and analyzed by IC.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 6. Supernate Concentrations of Total Base, Free Hydroxide, and Other Bases Excluding Carbonate for the As-Received Tank 51 SB6 Qualification Sample.

Species	Molarity	%RSD
Total Base	5.07	0.8
Free OH^-	3.53	0.7
Other Base Excluding CO_3^{2-}	1.06	5.5

Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 7. Decant B (Supernate) Analysis

	Unit	Result	%RSD
Supernate Density	g/mL	1.26	0.8
Fluoride	molar	<0.028	
Formate	molar	<0.012	
Chloride	molar	0.0137	2.9
Nitrite	molar	0.752	0.5
Bromide	molar	<0.0065	
Nitrate	molar	0.756	0.5
Phosphate	molar	<0.028	
Sulfate	molar	0.0980	1.0
Oxalate	molar	<0.0059	
Al	molar	0.484	2.3
B	molar	0.00347	2.6
Cr	molar	0.00311	1.8
K	molar	0.0113	3.1
Mo	molar	0.000296	2.9
Na	molar	6.09	1.9
P	molar	0.00396	2.3
S	molar	0.109	4.0
Si	molar	0.000474	0.5
Zn	molar	3.51E-05	9.2

Results are averages of four measurements. Anions are determined from IC, and elementals from ICP-AES.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 8. Decant C (Supernate) Analysis

	Unit	Result	%RSD
Supernate Density	g/mL	1.27	0.7
Fluoride	molar	<0.013	
Formate	molar	<0.0056	
Chloride	molar	<0.0072	
Nitrite	molar	0.498	0.3
Bromide	molar	<0.0032	
Nitrate	molar	0.493	0.3
Phosphate	molar	<0.0027	
Sulfate	molar	0.0676	0.7
Oxalate	molar	<0.0029	
Al	molar	0.355	0.5
B	molar	0.00243	0.6
Be	molar	5.89E-06	1.5
Ca	molar	6.18E-05	3.2
Cr	molar	0.00230	0.5
Fe	molar	2.13E-05	7.4
K	molar	0.00829	3.4
Mg	molar	1.94E-05	13
Mo	molar	2.21E-04	0.7
Na	molar	4.41	0.9
P	molar	0.00285	1.9
Pb	molar	4.39E-05	na
S	molar	0.0725	1.4
Si	molar	3.62E-04	7.4
Zn	molar	5.92E-05	1.4

Results are averages of four measurements. Anions are determined from IC, and elementals from ICP-AES.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 9. Post Decant C Slurry and Supernate Density and Weight Percent Solids

Property	Result	*%RSD*
Slurry Density	1.27	*0.7*
Supernate Density	1.20	*0.2*
Wt% Total Solids (Slurry Basis)	26.51	*0.2*
Wt% Dissolved Solids[a] (Supernate Basis)	21.56	*1.1*
Wt% Soluble Solids[b] (Slurry Basis)		
Wt% Insoluble Solids (Slurry Basis)	6.31	*NA*

Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 10. Post Decant C Aqua Regia Digestion of Total Solids

Element	Wt % of Total Solids	%RSD	Element	Wt % of Total Solids	%RSD
Al	5.98	4.2	Mg	0.0917	2.2
Ba	0.0390	2.5	Mn	1.75	2.1
Ca	0.242	2.6	Mo	0.00700	3.2
Cd	0.00321	4.8	Na	29.5	2.6
Ce	0.0582	2.2	Ni	0.677	2.8
Cr	0.0453	2.0	P	0.0718	20
Cu	0.0298	2.5	S	0.785	4.2
Fe	4.80	2.0	Si	0.206	4.0
Gd	0.0346	3.2	Sr	0.0182	2.5
Hg	2.05	4.2	Ti	0.00702	1.9
K	0.0949	1.3	U	1.11	3.3
La	0.0225	3.1	Zn	0.0182	3.7
Li	0.00768	3.5	Zr	0.0741	33

Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 11. Decant D (Supernate) Analysis

	Unit	Result	%RSD
Supernate Density	g/mL	1.15	0.7
Fluoride	molar	<0.014	
Formate	molar	<0.0060	
Chloride	molar	<0.0076	
Nitrite	molar	0.440	0.2
Bromide	molar	<0.0034	
Nitrate	molar	0.449	0.2
Phosphate	molar	<0.0030	
Sulfate	molar	0.0484	0.3
Oxalate	molar	<0.0032	
Al	molar	0.258	3.1
B	molar	0.00191	2.7
Ca	molar	6.11E-05	22
Cr	molar	0.00144	3.2
Fe	molar	7.05E-05	98
K	molar	0.00574	7.1
Mo	molar	1.65E-04	3.0
Na	molar	3.25	2.7
P	molar	0.00201	4.4
S	molar	0.0582	2.7
Zn	molar	2.60E-05	4.3

Results are averages of four measurements. Anions are determined from IC, and elementals from ICP-AES.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 12. Decant E (Supernate) Analysis

	Unit	Result	%RSD
Supernate Density	g/mL	1.12	0.4
Fluoride	molar	<0.0066	NA
Formate	molar	<0.0028	NA
Chloride	molar	<0.0035	NA
Nitrite	molar	0.262	3.5
Bromide	molar	<0.0016	NA
Nitrate	molar	0.285	3
Phosphate	molar	<0.0013	NA
Sulfate	molar	0.0421	14
Oxalate	molar	<0.0014	NA
Al	molar	0.190	1.6
B	molar	0.00144	0.5
Ca	molar	2.10E-05	4
Cr	molar	0.00124	1.4
K	molar	0.00477	0.4
Mo	molar	1.23E-04	3.1
Na	molar	2.45	1.4
P	molar	0.00154	2.4
S	molar	0.0488	2.0
Zn	molar	9.77E-06	14

Results are averages of four measurements. Anions are determined from IC, and elementals from ICP-AES.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 13. Decant F (Supernate) Analysis

	Unit	Result	%RSD
Supernate Density	g/mL	1.11	0.4
Fluoride	molar	<0.0064	NA
Formate	molar	<0.0027	NA
Chloride	molar	<0.0034	NA
Nitrite	molar	0.414	1.0
Bromide	molar	<0.0015	NA
Nitrate	molar	0.213	1.2
Phosphate	molar	<0.0013	NA
Sulfate	molar	0.0290	1.0
Oxalate	molar	<0.0014	NA
Al	molar	0.153	0.4
B	molar	0.00113	1.3
Ca	molar	4.46E-05	5.2
Cr	molar	0.00100	2.1
Fe	molar	2.74E-05	NA
K	molar	0.00369	8.9
Mo	molar	9.60E-05	2.9
Na	molar	2.14	0.6
P	molar	0.00121	4.6
S	molar	0.0368	3.8
Zn	molar	9.49E-06	7.9

Results are averages of four measurements. Anions are determined from IC, and elementals from ICP-AES.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 14. Post Decant F Slurry and Supernate Density and Weight Percent Solids

Property	Result	%RSD
lurry Density	1.17	1.2
upernate Density	1.11	0.4
Wt% Total Solids (Slurry Basis)	17.6	0.5
Wt% Calcined Solids (Slurry Basis)	13.2	2.7
Vt% Dissolved Solids Supernate Basis)	11.3	0.4
Wt% Soluble Solids (Slurry Basis)	10.5	NA
Wt% Insoluble Solids (Slurry Basis)	7.1	NA

Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 15. Post Decant F Aqua Regia Digestion of Total Solids

Element	Wt % of Total Solids	%RSD	Element	Wt % of Total Solids	%RSD
Al	7.81	2.2	Mg	0.144	1.3
Ba	0.0607	1.5	Mn	2.79	0.3
Ca	0.357	0.6	Mo	0.00670	3.5
Cd	0.00484	3.1	Na	24.9	0.3
Ce	0.0929	0.9	Ni	1.12	0.0
Co	0.00626	1.6	P	0.118	12
Cr	0.0441	1.6	Pb	0.0171	6.1
Cu	0.0464	1.1	S	0.663	4.9
Fe	7.76	0.8	Si	0.254	19.4
Gd	0.0550	0.7	Sr	0.0294	0.6
Hg	2.06	24	Ti	0.0111	1.2
K	0.0904	5.3	U	1.73	0.5
La	0.0360	2.5	Zn	0.0287	3.3
Li	0.0123	1.9	Zr	0.129	15

Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 16. Decant G (Supernate) Analysis

	Unit	Result	%RSD
Supernate Density	g/mL	1.08	0.3
Fluoride	molar	<0.0065	NA
Formate	molar	<0.0027	NA
Chloride	molar	<0.0035	NA
Nitrite	molar	0.392	6.4
Bromide	molar	<0.0015	NA
Nitrate	molar	0.236	4.9
Phosphate	molar	<0.0013	NA
Sulfate	molar	0.0219	0.8
Oxalate	molar	<0.0014	NA
Al	molar	0.117	1.4
B	molar	0.000812	0.6
Ca	molar	3.59E-05	3.6
Cu	molar	0.000726	0.5
La	molar	0.00266	7.2
Mo	molar	7.52E-05	1.5
Na	molar	1.62	1.6
P	molar	0.000740	1.7
S	molar	0.0272	0.4
Zr	molar	5.96E-06	4.6

Results are averages of four measurements. Anions are determined from IC, and elementals from ICP-AES.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 17. Decant H (Supernate) Analysis.

	Unit	Result	%RSD
Supernate Density	g/mL	1.06	0.3
Fluoride	molar	<0.0067	NA
Formate	molar	<0.0028	NA
Chloride	molar	<0.0036	NA
Nitrite	molar	0.297	1.8
Bromide	molar	<0.0016	NA
Nitrate	molar	0.144	1.7
Phosphate	molar	<0.0013	NA
Sulfate	molar	0.0174	2
Oxalate	molar	<0.0014	NA
Al	molar	0.0907	0.2
B	molar	0.000686	0.4
Ca	molar	7.00E-05	10
Cr	molar	0.000574	0.4
Fe	molar	1.31E-05	118
K	molar	0.00189	2.8
Mo	molar	5.62E-05	8.2
Na	molar	1.28	0.3
P	molar	0.000556	4.6
S	molar	0.0211	0.4

Results are averages of four measurements. Anions are determined from IC, and elementals from ICP-AES.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 18. Decant I (Supernate) Analysis

	Unit	Result	%RSD
Supernate Density	g/mL	1.06	0.6
Fluoride	molar	<0.0065	NA
Formate	molar	<0.0027	NA
Chloride	molar	<0.0035	NA
Nitrite	molar	0.237	2.6
Bromide	molar	<0.0015	NA
Nitrate	molar	0.124	2.7
Phosphate	molar	<0.0013	NA
Sulfate	molar	0.0149	2.2
Oxalate	molar	<0.0014	NA
Al	molar	0.0759	1.1
B	molar	0.000657	8.5
Ca	molar	0.0000986	25
Fe[†]	molar	0.0000230	NA
K	molar	0.00166	5.2
Mo	molar	0.0000448	5.2
Na	molar	1.08	0.9
P	molar	0.000461	2.9
S	molar	0.0178	2.5

Results are averages of four measurements. Anions are determined from IC, and elementals from ICP-AES.
[†] Reported result is for a single replicate; the other three replicates were reported as less than detectable.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 19. Densities and Weight Percent Solids for Tank 51 SB6 Qualification SRAT Receipt Sample

Property	Result (%RSD)
Slurry Density (g/mL)†	1.13 (1.5)
Supernate Density (g/mL) ‡	1.06 (0.6)
Wt% Total Solids (Slurry Basis)	15.1 (0.7)
Wt% Calcined Solids (Slurry Basis)	11.9 (0.8)
Wt% Dissolved Solids[a] (Supernate Basis)	5.80 (1.2)
Wt% Soluble Solids[b] (Slurry Basis)	5.23 (0.6)[c]
Wt% Insoluble Solids (Slurry Basis)	9.89 (0.7)[c]

† Temperature at time of slurry density measurements was 6 °C.
‡ Temperature at time of supernate density measurements was 7 °C.
[a] Also known as Uncorrected Soluble Solids
[b] Also known as Corrected Soluble Solids
[c] %RSD here is more correctly defined as % standard error for these calculated values
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 20. Concentrations of Elements in Total Dried Solids and Calcined Solids for the Tank 51 SB6 Qualification SRAT Receipt Sample.

Element	Wt% Total Solids	Wt% Calcine Solids	%RSD	Instrument	Prep Method
Ag	0.0138	0.0175	0.9	ICP-MS	AR
Al	11.0	13.9	0.5	ICP-AES	PF
As	<0.0010	<0.0013	-	AA	AR
B	<0.0068	<0.0087	-	ICP-AES	AR
Ba	0.0917	0.117	1.3	ICP-AES	AR/PF
Be	<0.00074	<0.00094	-	ICP-AES	AR
Ca	0.527	0.670	0.9	ICP-AES	AR
Cd	0.00799	0.0102	4.5	ICP-AES	AR/PF
Ce	0.139	0.177	0.7	ICP-MS	AR
Co	0.00869	0.0110	1.8	ICP-AES	AR
Cr	0.0448	0.0570	3.2	ICP-AES	AR/PF
Cu	0.0711	0.0903	2.3	ICP-AES	AR/PF
Fe	11.8	14.9	1.4	ICP-AES	AR/PF
Gd	0.0823	0.105	3.2	ICP-MS	AR
Hg	3.12	-	6.7	CVAA	AR
K	0.0595	0.0756	5.4	ICP-AES	AR
La	0.0713	0.0907	1.2	ICP-MS	AR
Li	0.0172	0.0218	2.0	ICP-AES	AR
Mg	0.214	0.272	1.5	ICP-AES	AR/PF
Mn	4.15	5.28	0.9	ICP-AES	AR/PF
Mo	0.00514	0.00653	5.6	ICP-AES	AR
Na	15.4	19.6	0.9	ICP-AES	AR
Nd	0.258	0.328	0.3	ICP-MS	AR
Ni	1.69	2.15	1.2	ICP-AES	AR/PF
P	0.143	0.181	7.2	ICP-AES	PF
Pb	0.0158	0.0201	4.2	ICP-MS	AR
S	0.375	0.477	4.7	ICP-AES	AR
Se	<0.0021	<0.0026	-	AA	AR
Sb	<0.014	<0.018	-	ICP-AES	AR
Si	0.711	0.904	2.8	ICP-AES	PF
Sn	<0.0081	<0.010	-	ICP-AES	AR
Sr	0.0431	0.0548	1.8	ICP-AES	AR/PF
Ti	0.0164	0.0208	4.6	ICP-AES	AR/PF
Th	2.98	3.78	1.6	ICP-MS	AR
U	2.33	2.97	1.1	ICP-MS	AR
V	<0.0046	<0.0058	-	ICP-AES	AR
Zn	0.0413	0.0525	8.0	ICP-AES	AR/PF
Zr†	0.156	0.198	2.1	ICP-AES	AR

Results are averages of dissolution and analysis of four or eight aliquots of the SB6 slurry.
† Reported result is the average of three replicates.
ICP-MS ≡ inductively coupled plasma – mass spectrometry, ICP-AES ≡ inductively coupled plasma – atomic emission spectroscopy, CVAA ≡ cold vapor atomic absorption spectroscopy, AA ≡ Atomic Absorption, AR ≡ aqua regia digestion, PF ≡ peroxide fusion digestion.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 21. Concentrations of Noble Metals and Silver in Total Dried Solids for the SRAT Receipt Tank 51 SB6 Qualification Sample.

Element	Wt% Total Solids	%RSD
Ru	0.0924	0.2
Rh	0.0187	3.4
Pd	0.00304	0.5
Ag	0.0138	0.9

Results are Averages of Aqua Regia Digestions and ICP-MS Analyses of Four SB6 Slurry Aliquots
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 22. Concentrations of Anions and Cations on a Slurry Basis for the SRAT Receipt Tank 51 SB6 Qualification Sample

Anion	mg/kg slurry	%RSD
Br^-	<100	-
Cl^-	<100	-
F^-	<100	-
HCO_2^-	100	7.8
NO_3^-	6,840	1.2
NO_2^-	10,000	0.7
$C_2O_4^{2-}$	<100	-
PO_4^{3-}	<100	-
SO_4^{2-}	1200	0.8

Results are averages of four SB6 slurry aliquots.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 23. Slurry Concentrations of Total Base, Free Hydroxide, and Other Bases Excluding Carbonate for the SRAT Receipt Tank 51 SB6 Qualification Sample

Species	Mol/L Slurry	%RSD
Total Base	0.58	4.8
Free OH^-	0.40	1.8
Other Base Excluding CO_3^{2-}	0.12	8.9

Results are averages of four SB6 slurry aliquots.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 24. Slurry and Supernate Concentrations of TIC/TOC for the SRAT Receipt Tank 51 SB6 Qualification Sample. .

Species	mg/kg slurry	%RSD	mg/L supernate	%RSD
Total Inorganic Carbon	913	16	910	5.5
Total Organic Carbon	483	71	<70	-
Total Carbon	1400	19	910	5.5

Results are averages of four SB6 slurry aliquots and three SB6 supernate aliquots.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 25. Weight Percent Solids, Density, and pH of the SB6 Qualification SRAT Product

Property	Result	%RSD
Weight % Total Solids (slurry basis)	25.9	0.2
Weight% Dissolved Solids (supernate basis)	9.35	0.3
Weight % Insoluble Solids (slurry basis)	18.3	NA
Weight% Soluble Solids (slurry basis)	7.6	NA
Weight % Calcined Solids (slurry basis)	17.7	0.4
Slurry Density (g/mL)	1.23	0.8
Supernate Density (g/mL)	1.08	3
pH	10	NA

Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 26. Concentrations of Anions, Amonium, and Carbon on a Slurry Basis for the SB6 Qualification SRAT Product

Analyte	mg/kg slurry	%RSD
Fluoride	<1,000	NA
Formate	43,600	1%
Chloride	<1,000	NA
Nitrite	<1,000	NA
Nitrate	26,200	1%
Phosphate	<100	NA
Sulfate	2,370	6%
Oxalate	<100	NA
Ammonium	<200	NA
Total Carbon †	17,400	NA
Total Inorganic Carbon	1,800	2
Total Organic Carbon	15,600	0.8

Results are averages of four SB6 slurry aliquots.
† Total Carbon = Total Inorganic Carbon + Total Organic Carbon
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 27. Physical Properties of As-Made SB6 SME Product (Prior to Dilution to 45 wt % Total Solids)

Property	Result	% RSD	# of Replicates
Wt % Total Solids	52.5	0.4%	4 replicates
Wt % Calcined Solids	46.9	NA	1 replicate
Calcine Factor (mass oxides/mass total solids)	0.89	NA	NA
Wt % Dissolved Solids	NA – SME product was too thick and sticky for adequate measurement.		
Wt % Insoluble Solids			
Wt % Soluble Solids			
Supernate Density			

Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 28. SB6 SME Product (at 45 wt % Total Solids) Density, Anion, and Cation Concentrations

Measurement	Result (mg/kg slurry)	% RSD	# of Replicates
Density, T = 21 °C	1.42	NA	1 replicate †
Fluoride	<50	NA	-
Formate	26,300	2	4 replicates
Chloride	<50	NA	-
Nitrite	613	2	-
Bromide	<50	NA	-
Nitrate	15,500	1	4 replicates
Phosphate	<300	NA	-
Sulfate	1,450	2	-
Oxalate	147	8	-
Ammonium	<2,500	NA	-

† Because of the stickiness of the sample and difficulty in removing it from the density measurement container, the density measurement was not repeated. It should be noted that the measured density is similar to what one would expect for a ~ 50 wt % total solids SME product.
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Table C - 29. SB6 SME Product (at 45 wt % Total Solids) Carbon Analysis

Analyte	Average (mg/kg slurry)	% RSD	# of Replicates
Total Inorganic Carbon	1,800	7	3
Total Organic Carbon	10,200	1	4
Total Carbon	12,000 †	NA	NA

† Total Carbon = Total Inorganic Carbon + Total Organic Carbon
Values in the %RSD column are relative to the true calculated averages of the quantities in the tables, while the average values reported have been rounded off to a reasonable number of significant figures.

Appendix D. Supplemental PCT Data

Table D - 1. PCT Elemental Releases for ARM, EA and the SB6 Qualification Glass

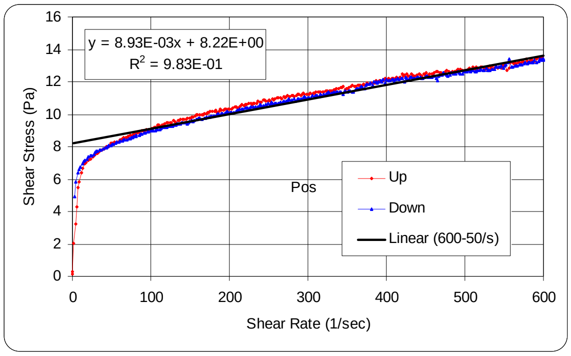
Glass ID	Elemental Release - Dilution Factor Corrected (ppm)						
	Li	B	Na	Si	Al	Fe	U
ARM-1-P83	13.076	15.906	35.385	58.301	4.853	<0.071	<2.996
ARM-2-P85	13.506	16.975	37.048	59.950	4.883	<0.071	<2.994
ARM-3-P86	12.806	15.637	34.711	57.290	4.852	<0.071	<2.996
EA-1-P72	196.785	602.821	1677.682	907.297	<3.842	<0.429	<18.166
EA-2-P74	192.660	594.529	1654.876	892.816	<3.841	<0.429	<18.163
EA-3-P76	199.053	637.623	1771.857	940.085	<3.842	<0.429	<18.168
SB-6 GLS-1-P77	19.583	9.977	91.343	118.982	20.392	6.404	4.382
SB-6 GLS-2-P80	20.041	10.273	93.301	119.910	20.311	5.254	4.379
SB-6 GLS-3-P81	19.403	9.870	90.279	115.206	19.942	5.491	4.177
SB-6 GLS-4-P82	20.311	10.442	94.312	120.584	20.445	5.288	4.513

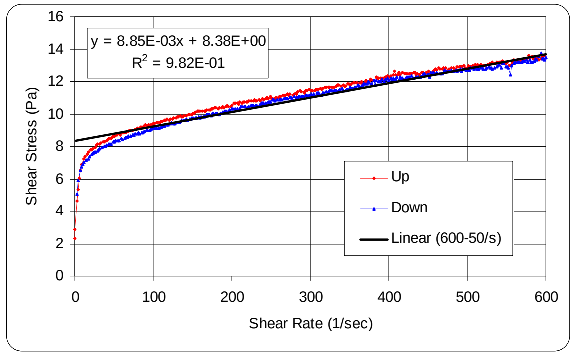
Appendix E. Settling Data

Decant A						Decant E				
Date	Time	Level (mL)	Setling time (d)	Relative level		Date	Time	Level (mL)	Setling time (d)	Relative level
10/13/2009		2700	0.0	1		12/7/2009	16:50	3300	0.0	1
10/15/2009		2600	2.0	0.962962963		12/8/2009	7:30	3225	0.6	0.977272727
10/16/2009		2560	3.0	0.948148148		12/8/2009	12:25	3000	0.8	0.909090909
10/17/2009		2500	4.0	0.925925926		12/8/2009	17:00	2930	1.0	0.887878788
10/18/2009		2440	5.0	0.903703704		12/9/2009	7:10	2800	1.6	0.848484848
10/19/2009		2400	6.0	0.888888889		10-Dec	7:05	2610	2.6	0.790909091
10/20/2009		2390	7.0	0.885185185		11-Dec	6:50	2400	3.6	0.727272727
10/21/2009		2380	8.0	0.881481481						
10/22/2009		2370	9.0	0.877777778		Decant F				
10/23/2009		2290	10.0	0.848148148		Date	Time	Level (mL)	Setling time (d)	Relative level
10/26/2009		2190	13.0	0.811111111		12/11/2009	13:30	3300	0.0	1
10/27/2009		2175	14.0	0.805555556		12/14/2009	8:00	2550	2.8	0.772727273
10/28/2009		2120	15.0	0.785185185		12/15/2009	7:00	2350	3.7	0.712121212
10/29/2009		2075	16.0	0.768518519						
10/30/2009		2050	17.0	0.759259259		Decant G				
10/31/2009		2030	18.0	0.751851852		Date	Time	Level (mL)	Setling time (d)	Relative level
11/1/2009		2000	19.0	0.740740741		12/15/2009	13:55	3250	0.0	1
11/2/2009		1990	20.0	0.737037037		12/16/2009	10:30	2950	0.9	0.907692308
						12/16/2009	16:00	2850	1.1	0.876923077
Decant B						12/17/2009	7:00	2625	1.7	0.807692308
Date	Time	Level (mL)	Setling time (d)	Relative level		12/21/2009	7:00	2000	5.7	0.615384615
11/5/2009	15:00	3450	0.0	1						
11/6/2009	8:00	3300	0.7	0.956521739						
11/7/2009	14:00	3100	2.0	0.898550725		Decant H				
11/9/2009	6:40	2925	3.7	0.847826087		Date	Time	Level (mL)	Setling time (d)	Relative level
11/10/2009	7:00	2795	4.7	0.810144928		12/21/2009	13:55	3250	0.0	1
11/11/2009	7:00	2760	5.7	0.8		12/22/2009	8:00	2850	0.8	0.876923077
11/12/2009	7:00	2610	6.7	0.756521739		12/23/2009	6:50	2700	1.7	0.830769231
11/13/2009	7:00	2500	7.7	0.724637681		12/23/2009	16:45	2550	2.1	0.784615385
11/14/2009	13:00	2400	8.9	0.695652174		12/26/2009	14:00	2010	5.0	0.618461538
11/16/2009	5:20	2300	10.6	0.666666667		12/28/2009	7:00	1975	6.7	0.607692308
Decant C										
Date	Time	Level (mL)	Setling time (d)	Relative level		Decant I				
11/18/2009	15:45	3450	0.0	1		Date	Time	Level (mL)	Setling time (d)	Relative level
11/19/2009	16:00	3200	1.0	0.927536232		12/28/2009	14:45	3250	0.0	1
11/20/2009	13:00	2900	1.9	0.84057971		12/29/2009	8:00	2950	0.7	0.907692308
11/21/2009	12:00	2810	2.8	0.814492754		12/29/2009	16:00	2800	1.1	0.861538462
11/23/2009	8:45	2620	4.7	0.75942029		12/30/2009	6:30	2600	1.7	0.8
11/24/2009	6:50	2550	5.6	0.739130435		12/30/2009	16:30	2480	2.1	0.763076923
11/25/2009	9:50	2400	6.8	0.695652174		12/31/2009	11:00	2250	2.8	0.692307692
11/27/2009	13:00	2230	8.9	0.646376812		1/2/2010	13:00	2000	4.9	0.615384615
11/28/2009	13:00	2200	9.9	0.637681159		1/4/2010	8:10	1975	6.7	0.607692308
11/30/2009	6:00	2125	11.6	0.615942029		1/5/2010	8:30	1900	7.7	0.584615385
						1/6/2010	5:30	1900	8.6	0.584615385
Decant D						1/7/2010	9:30	1900	9.8	0.584615385
Date	Time	Level (mL)	Setling time (d)	Relative level						
12/1/2009	11:50	3200	0.0	1						
12/2/2009	7:05	3000	0.8	0.9375						
12/3/2009	14:50	2820	2.1	0.88125						
12/4/2009	12:50	2600	3.0	0.753623188						
12/7/2009	12:15	2250	6.0	0.652173913						

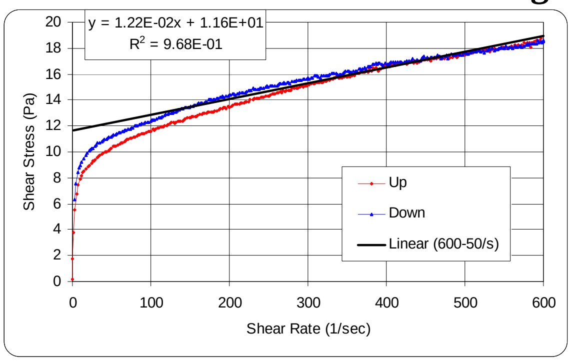
Appendix F. Rheology Flow Curves

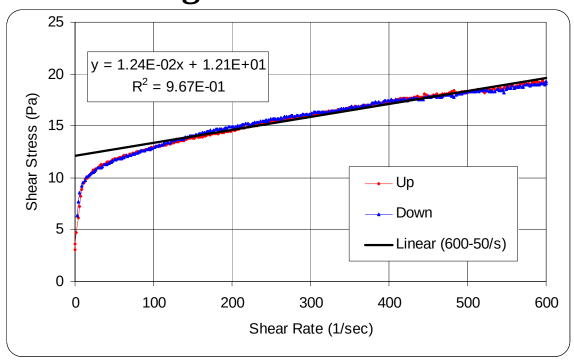
SRAT Receipt



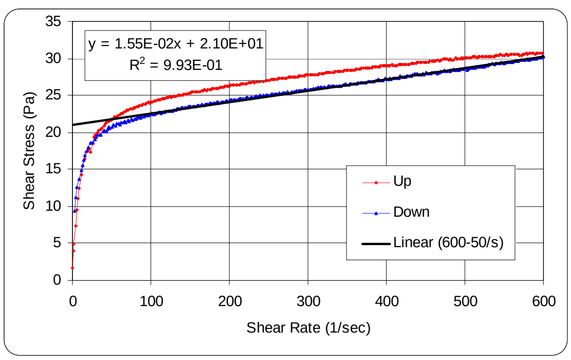


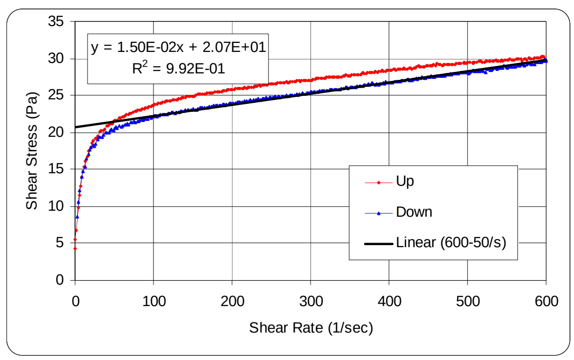
SRAT Material Following Caustic Boiling/Concentration





SRAT Product





Distribution:

A. B. Barnes, 999-W
D. A. Crowley, 773-43A
S. D. Fink, 773-A
B. J. Giddings, 786-5A
C. C. Herman, 999-W
S. L. Marra, 773-A
F. M. Pennebaker, 773-42A
C. J. Bannochie, 773-42A
J. M. Gillam, 766-H
B. A. Hamm, 766-H
J. F. Iaukea, 704-30S
A. V. Staub, 704-27S
J. E. Occhipinti, 704-S
D. K. Peeler, 999-W
J. W. Ray, 704-S
H. B. Shah, 766-H
D. C. Sherburne, 704-S
M. E. Stone, 999-W
M. A. Broome, 704-29S
R. N. Hinds, 704-S
J. P. Vaughan, 773-41A
J. M. Bricker, 704-27S
T. L. Fellinger, 704-26S
E. W. Holtzscheiter, 704-15S
M. T. Keefer, 766-H